



Identification and Quantitative Measurements of Chemical Species by Mass Spectrometry

Mark A. Zondlo and David S. Bomse
Southwest Sciences, Inc., Santa Fe, New Mexico

The NASA STI Program Office . . . in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program Office plays a key part in helping NASA maintain this important role.

The NASA STI Program Office is operated by Langley Research Center, the Lead Center for NASA's scientific and technical information. The NASA STI Program Office provides access to the NASA STI Database, the largest collection of aeronautical and space science STI in the world. The Program Office is also NASA's institutional mechanism for disseminating the results of its research and development activities. These results are published by NASA in the NASA STI Report Series, which includes the following report types:

- **TECHNICAL PUBLICATION.** Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA's counterpart of peer-reviewed formal professional papers but has less stringent limitations on manuscript length and extent of graphic presentations.
- **TECHNICAL MEMORANDUM.** Scientific and technical findings that are preliminary or of specialized interest, e.g., quick release reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.
- **CONTRACTOR REPORT.** Scientific and technical findings by NASA-sponsored contractors and grantees.

- **CONFERENCE PUBLICATION.** Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or cosponsored by NASA.
- **SPECIAL PUBLICATION.** Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.
- **TECHNICAL TRANSLATION.** English-language translations of foreign scientific and technical material pertinent to NASA's mission.

Specialized services that complement the STI Program Office's diverse offerings include creating custom thesauri, building customized databases, organizing and publishing research results . . . even providing videos.

For more information about the NASA STI Program Office, see the following:

- Access the NASA STI Program Home Page at <http://www.sti.nasa.gov>
- E-mail your question via the Internet to help@sti.nasa.gov
- Fax your question to the NASA Access Help Desk at 301-621-0134
- Telephone the NASA Access Help Desk at 301-621-0390
- Write to:
NASA Access Help Desk
NASA Center for Aerospace Information
7121 Standard Drive
Hanover, MD 21076



Identification and Quantitative Measurements of Chemical Species by Mass Spectrometry

Mark A. Zondlo and David S. Bomse
Southwest Sciences, Inc., Santa Fe, New Mexico

Prepared under Contract NAS3-01164

National Aeronautics and
Space Administration

Glenn Research Center

Trade names or manufacturers' names are used in this report for identification only. This usage does not constitute an official endorsement, either expressed or implied, by the National Aeronautics and Space Administration.

Available from

NASA Center for Aerospace Information
7121 Standard Drive
Hanover, MD 21076

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22100

Available electronically at <http://gltrs.grc.nasa.gov>

IDENTIFICATION AND QUANTITATIVE MEASUREMENTS OF CHEMICAL SPECIES BY MASS SPECTROMETRY

Final

Mark A. Zondlo and David S. Bomse
Southwest Sciences, Inc.
Santa Fe, NM 87505

1. Background

Mass spectrometry is often known as the gold standard of analytical chemistry techniques. No other method can provide a similar combination of general utility, response time, and detection sensitivity. But, mass spectrometers have typically been large, heavy, and required large amounts of power to operate.

In 1999, Southwest Sciences began working with Mass Sensors (St. Louis, MO) to develop a miniaturized version of a double-focusing type of mass spectrometer. Much of that work was supported by NASA's SBIR program. The analyzer portion of the Mass Sensors device – which was, in turn, based on intellectual property licensed from the University of Minnesota – is small enough to fit in the palm of one hand. It uses a combined magnetic and electrostatic fields to generate mass spectra; the magnetic field acts as a momentum selector (which is actually a mass analyzer) while the electrostatic field acts as an energy analyzer. Careful selection of the relative strengths of the two types of fields improves mass resolution by reducing the effects of the distribution of the ion kinetic energies. Although double-focusing mass spectrometers had been known for nearly 50 years, the key innovation in the Mass Sensors instrument was a novel method for constructing the elements used to generate the electrostatic field that nearly eliminated field distortion at the sides of the mass analyzer. This made it possible to shrink the analyzer so that ion trajectories filled nearly the entire open portion of the analyzer. Previous designs required the analyzer to be substantially large than the ion path cross section in order to guarantee uniform fields along the ion trajectories.

The SBIR-funded work at Southwest Sciences showed the instrument to operate well at ion masses from about 15 to 110 daltons. The mass spectral peaks had a Gaussian shape with a fixed $m/\Delta m$ width that allowed straightforward deconvolution of overlapping mass peaks. The mass spectrometer was equipped with a newly designed ion multiplier (Detector Technologies, Inc.) that allowed operation at sample pressures up to 10^{-4} torr, or an order of magnitude larger than is possible with conventional ion multipliers. This higher pressure operation further increased detection sensitivity by increasing the number density of analyte gas in the ion source region.

The initial success of Southwest Sciences' work with the Mass Sensors mass spectrometer motivated the current project. A number of NASA programs – particularly space-borne and microgravity drop tower experiments – would benefit from the availability of a compact, truly portable, mass spectrometer. Potential applications ranged from monitoring crew physiology on board the ISS by measuring gases in exhaled breath to detecting reaction intermediates during microgravity combustion. Despite the successes of the SBIR-funded work, Mass Sensors was unable to commercialize their mass spectrometer technology. Nearly all of the instruments suffered from performance degradation – diminished ion signals, reduced mass resolution, or both – on a time scale ranging from several hours to several days. This problem was attributed to deposition onto the inner surface of the mass analyzer of pyrolysis products formed at the hot wire filament in the ionizer. Performance could be restored by cleaning the analyzer, but the problem would recur on the same short time scale. In some ways, the biggest unresolved question was why the instrument that was used at Southwest Sciences in the Phase II SBIR project performed as well as it did for such an extended time.

Given their inability to produce reliable commercial instruments, Mass Sensors was unable to bring products to market and, ultimately, terminated their business in February 2004.

When it became clear that Mass Sensors would not be able to meet the requirements of this project, Southwest Sciences turned to other sources of miniature mass spectrometers. Several vendors were offering devices intended, for the most part, as benchtop analytic instruments and used quadrupole, time-of-flight, or ion trap technology. We selected the quadrupole mass spectrometer manufactured by Ferran Scientific (San Diego, CA). Ferran's technology was truly innovative. The mass analyzer consists of a 4×4 array of quadrupole rods that form a set of 9 (a 3×3 array) matched quadrupole filters operated in unison. The filter sits between a single ionization source and Faraday cup detector that serve all nine quadrupole sections. The entire assembly – ion source, quadrupole array, and Faraday cup – fits within a 12 cm^3 vacuum enclosure. An electronics head, attached to the vacuum enclosure, measures $4.7 \times 6.0 \times 5.6 \text{ cm}$ and weighs 100 g. The electronics base unit (computer network interface or CNI in Ferran's syntax) is $5.9 \times 14.0 \times 16.1 \text{ cm}$, weighs 750 gm, and requires 1.5 A at 24 V DC.

We selected the Ferran device over other newly developed quadrupole mass spectrometers (such as the Stanford Research and Infinicon instruments) because the Ferran is smaller, can operate at higher pressures, and uses less power. Similar concerns – particular the operating pressure and electronics – helped rule out the ion traps and time-of-flight mass spectrometers. As mass spectrometers get smaller, vacuum requirements become more important because the size, weight, and power needs of the vacuum pumps exceed those of the mass spectrometers. For example, the miniaturized cylindrical ion trap mass spectrometer developed by Prof. Graham Cooks at Purdue University is roughly 1 cm^3 , but the entire instrument including pumps, electronics, and computer weighs 16 kg.[†] Much of the weight is due to the vacuum requirements.

[†]See G. E. Patterson *et al.*, "Miniature Cylindrical Ion Trap Mass Spectrometer," *Analytical Chemistry* **2002**, 74, 6145-6153 and L. S. Ritter *et al.*, "Analytical Performance of a Miniature Cylindrical Ion Trap Mass Spectrometer," *Analytical Chemistry* **2002**, 74, 6154-6162.

2. Physical Description of the Delivered System

The completed mass spectrometer system is a fully-contained, automated instrument consisting of a needle sampling inlet, a custom-designed, small-scale gas chromatograph and a miniature, quadrupole mass spectrometer. The system is mounted on a two-tier, anodized aluminum breadboard having overall dimensions 38×45.5×30.5 cm, Figs. 1 and 2. The upper tier supports the sample control valves, chromatograph, mass spectrometer and controller, and turbomolecular pump. There is also space for a lecture bottle of helium to be used as the chromatograph carrier gas. The bottom tier contains a roughing pump and control electronics for the sampling inlet, chromatograph heater, multiport valve, and turbomolecular pump. A laptop computer that controls the instrument and collects data is also included.

The instrument operates using 120 VAC. Two uninterruptible power supplies (UPS) are included so that the user has a choice between operating time and UPS size and weight.

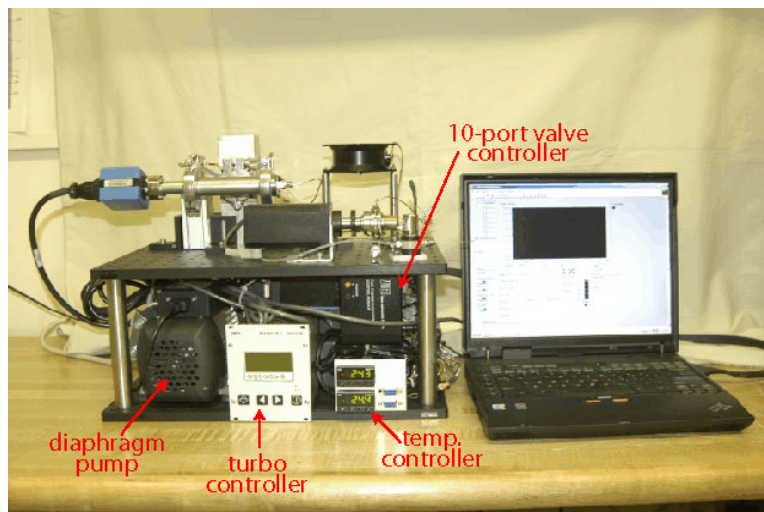


Figure 1. Photograph of the experimental setup. The top plate contains the sampling system (column, needle, valves) and turbomolecular pump while the lower plate contains the electronics, controllers, and diaphragm pump. The entire assembly plugs into one 120 V AC outlet.

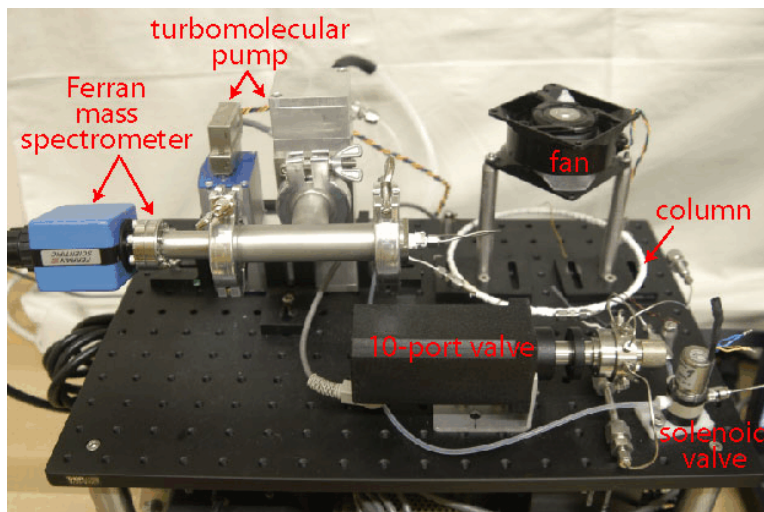


Figure 2 - Photograph of the upper platform of the system showing the mass spectrometer, vacuum system, and gas chromatograph. There is room also for a carrier gas lecture bottle.

3. Operation of the Delivered System

3.1 Mass Spectrometer

The Ferran Scientific Symphony quadrupole mass spectrometer, billed by the vendor as “the world’s smallest mass spectrometer,” consists of three components. The micropole array (MPA) is the actual mass spectrometer that sits inside the vacuum system. It is a miniature array of quadrupole mass spectrometers designed to operate simultaneously (in parallel). Atoms and molecules are ionized in a single Nier-type ion source following electron impact (EI). Electrons are emitted from hot filaments located outside the field of view (main axis) of the quadrupole detector. Using electrostatic lenses, ions are extracted, focused, and injected into individual quadrupole mass filters. Ions with a specific mass-to-charge ratio are filtered by applying a combination of DC and RF voltages of the poles forming a quadrupole of the array. Filtered ions in each quadrupole are collected on individual Faraday type detectors. All individual detectors contribute to generate a single current signal. A mass spectrum is generated by scanning both AC and DC voltages applied on the poles of the Micropole. The MPA chassis includes a total of 16 rods and 11 electrical pins all secured in a glass-to-metal structure and sealed in a high temperature oven. Operation at high temperature avoids the type of contamination that plagued the Mass Sensors design.

The electrostatic lenses, apertures, and plates are made of photo-etched parts. The sensor construction does not use ceramic parts to avoid the type of patch charging effects that are common with other quadrupole mass spectrometers.

The Ferran quadrupole can operate at one of two ionization settings: 70 eV and 43 eV. All of the development work was done using 70 eV ionization.

The second part of the Ferran mass spectrometer is the spectra converter (SC), the blue box that attaches to the MPA and is shown in Fig. 3. The SC includes a pre-tuned RF power supply and an electrometer. Using a built-in guide pin, the SC is easily connected and secured to the back of the MPA with a connector latch. The combination can be mounted in any orientation. Ferran offers three different sizes of MPA and three different spectra converters. Specifications for the allowed combinations of the micropoles and spectra converters are listed in Table 1. The combination used in the deliverable is highlighted; it was selected to give the best combination of mass range and resolution.

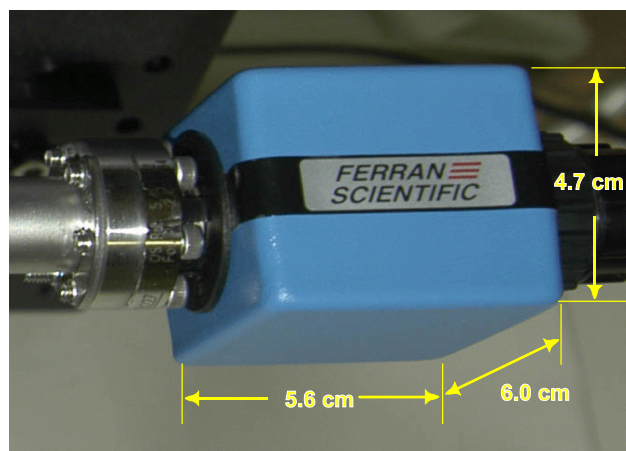


Figure 3 - Detail photograph of the spectra converter (SC) on the Ferran Scientific quadrupole mass spectrometer.

Table 1 - Ferran Scientific Quadrupole Mass Spectrometers

MPA	MPA6-1-2/45	MPA6-11-2/45	MPA6-5-2/6	MPA6-7-2/65	MPA6-5-2/100	MPA6-1-4/300
SC	SC6-18	SC6-18	SC6-14	SC6-14	SC6-11	SC6-07
mass range	2-45	2 - 45	2 - 65	2 - 65	2 - 100	4 - 300
resolution (FWHM)	0.5	1.5	0.8	0.9	1.0	1.5
P_{max} (torr)	1×10^{-3}	1×10^{-2}	5×10^{-3}	7×10^{-3}	5×10^{-3}	1×10^{-3}

The third portion of the mass spectrometer, the Computer Network Interface (CNI), Fig. 4, includes the power supplies, the control circuitry and the communications board. All circuits are driven by an internal microcomputer and embedded control software (firmware). The CNI connects to a PC via RS-232, RS-485, or TCP/IP serial interface, and to the SC using a shielded cable. The CNI (and the rest of the mass spectrometer) is powered by 24 V DC at up to 1.5 A.

An RS-232 serial interface is used in the deliverable for communication between the host laptop computer and the CNI. The settings are 9800 baud, 8 data bits, no parity, 1 stop bit, and XON/OFF flow control. Data strings are terminated by a line feed character (decimal 13). Communications with the CNI entail reading and/or writing a set of registers.



Figure 4 - Computer Network Interface (CNI) for the Ferran Scientific quadrupole mass spectrometer.

3.2 Gas Chromatograph

The custom gas chromatograph uses a micropacked, HayeSepQ, Silcosteel® column from Restek (2 m long \times 1 mm i.d. \times 1.59 mm o.d.). Silcosteel columns are composed of stainless steel on the outside but contain an inner coating of silica. The silica coating results in fewer adsorption problems relative to a stainless steel surface. The stainless steel exterior provides more rigidity and prevents column breakage, traits particularly desirable for drop tower and space-borne environments. The Silcosteel column contains 100/120 mesh adsorbents of HayeSepQ, an adsorbent that shows excellent separation for many organic species. The fine mesh allows for a large number of theoretical plates, thereby allowing greater separation of species eluting from the column. We have also used zero dead volume fittings where possible. Figure 5 shows chromatographs of mixtures of alkanes (top) and alkenes (bottom) in helium carrier gas.

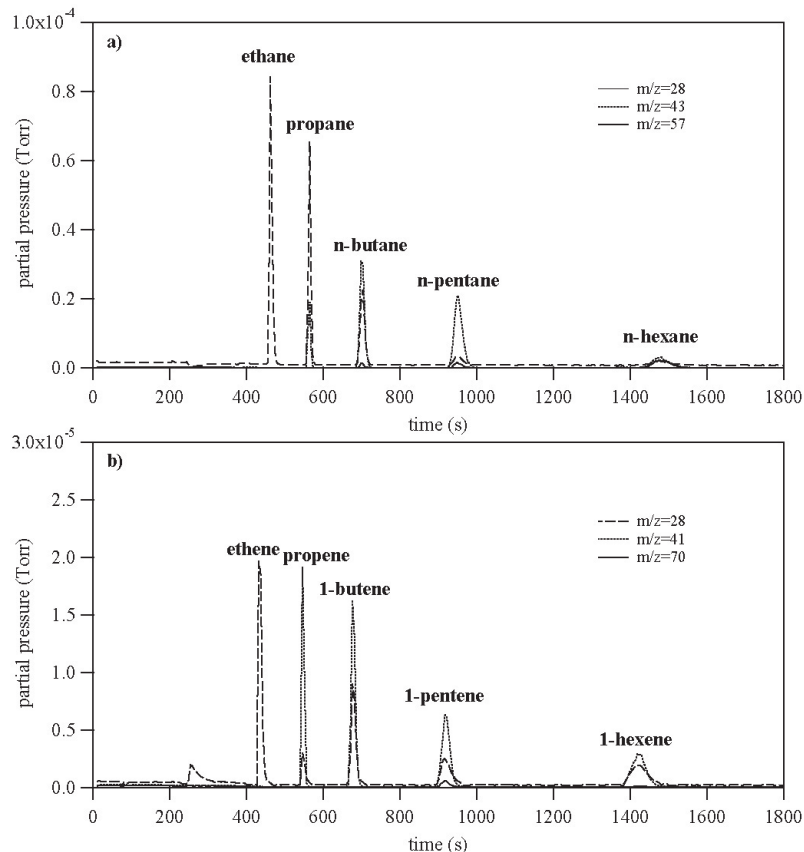


Figure 5 - Chromatograms of 1000 ppm calibration mixtures of alkanes (top) and alkenes (bottom) in helium.

The column is wrapped with thin (0.20 mm diameter), polyimide-insulated nichrome wire. By passing a current through the nichrome wire, the column can be resistively heated in a fast manner. The total resistance of the wire used to wrap the column is 452 Ohms, and thus currents of 0.25 A are used. The column is supported 3 cm above the top plate by three wires attached to posts. A fan was placed 12 cm directly above the center of the column for efficient and uniform cooling. Figure 6 shows a plot of the temperature response for warming the

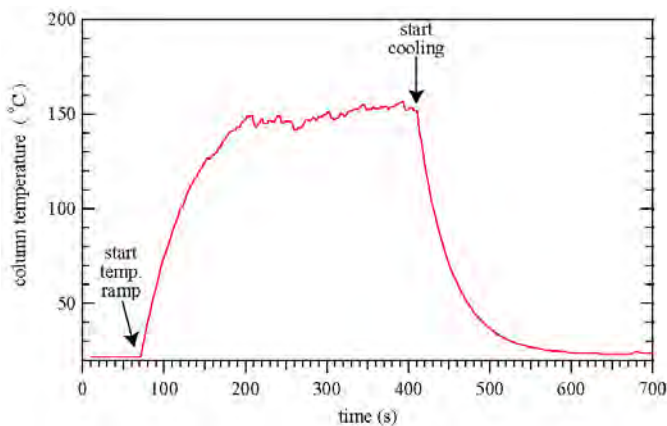


Figure 6 - Response of the column temperature to rapid warming and cooling.

column from ambient temperatures to 155 °C starting at 70 s. The initial heating rate is $\sim 90^{\circ}\text{C min}^{-1}$.

The low thermal mass of the wire also allows for rapid cooling. At 410 s the heating stops, the fan is started, and an exponential temperature decay is observed with a time constant of ~ 40 s. Cooling the column to near ambient temperatures occurs within three minutes. Overall, the duty cycle due to heating and cooling of the column is ~ 10 minutes.

The column heater control and temperature monitoring are performed by a pair of PID controllers (Omega CNi3222-C24). Two controllers are used because readout of the current temperature is not possible during programmed temperature ramp. Hence, the second controller simply provides a measurement of the instantaneous temperature.

Gas sampling and introduction to the chromatographic column are controlled by a pair of commercial, computer-driven valves, Fig. 7. Samples are drawn through a needle into a small reservoir using a three-port PEEK valve. The valve normally connects the reservoir to the vacuum pump so that the reservoir remains evacuated until a sample is acquired. Then, the valve switches to admit gas through the sampling needle into the reservoir. Flow rates for typical sample durations are limited by the conductance of the needle. PEEK was chosen because of its excellent chemical and thermal stability[†]. We found PEEK valves to leak under vacuum; but this is not a problem for the sample inlet that is pumped continuously. In this case, the chemical properties of the valve are more important than its vacuum characteristics.

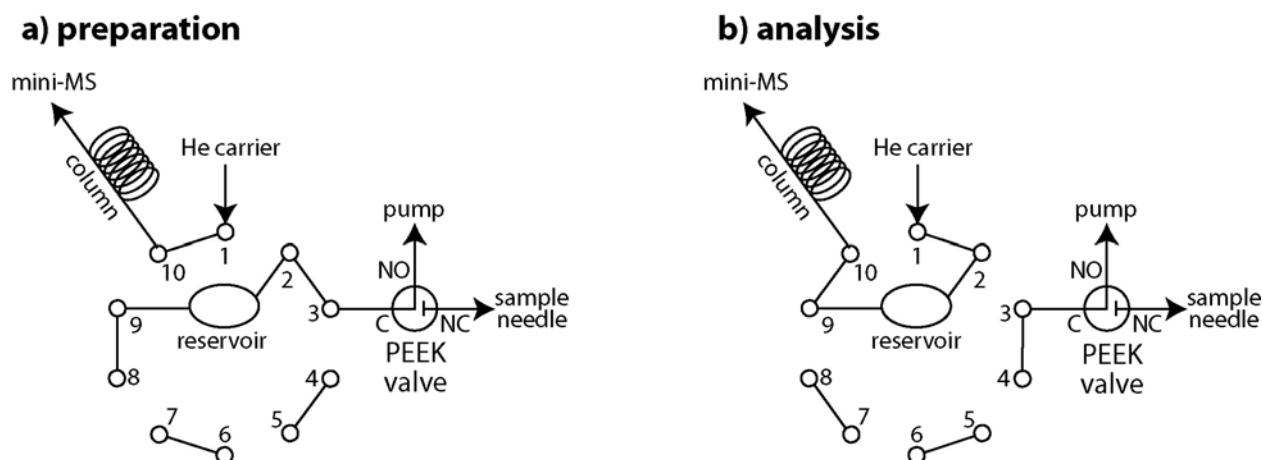


Figure 7 - Computer controlled valves used for sampling acquisition and for injecting the sampled gas onto the chromatographic column.

A ten-port, two-position, stainless steel and Nitronic 60 valve from Valco (P/N ET2C10UWT) is composed of stainless steel, has ten ports for 1/16" tubing, and is used to direct the sample from the reservoir to the chromatographic column. The valve is controlled by a 24 V DC microelectric actuator that can be switched by a 5 V logic signal from the computer. The valve can withstand up to 300 psi gas pressure and temperatures up to 330°C. The bores inside the valve are the same size

[†]See the Victrex web site: <http://www.victrex.com/>.

(0.75 mm) as the inner diameter of the tubing leading to the ports, thereby minimizing the amount of dead volume in the sampling regime. Although not all ten ports are needed for the current instrument design, the availability of additional ports for only a slight increase in cost allows for greater adaptability in future applications as well as for redundancy if any of the ports become unuseable in the future.

3.3 Vacuum System

The delivered mass spectrometer system is equipped with a Pfeiffer Vacuum model TPD 011 turbomolecular pump backed by a Pfeiffer MVP 015-2 diaphragm pump. The turbomolecular pump and controller are visible in the photograph in Fig. 2. The pump is one of a new generation of small, lightweight turbo pumps, and has a nominal pumping speed of 10 liters sec^{-1} for N_2 and 6 liters sec^{-1} for He with an ultimate pressure of better than 5×10^{-5} mbar (4×10^{-5} torr). It can tolerate a backing pressure of up to 25 mbar (19 torr). Compression ratios are 3×10^6 and 3×10^3 for N_2 and He, respectively. The pump requires 1 A at 12 V DC and needs no external cooling. It weighs 1.8 kg.

One end of the diaphragm pump is visible in the photograph in Fig. 2. Rough outer dimensions for the MVP 015-2 diaphragm pump are $29 \times 17 \times 17$ cm. The pump weighs 6.5 kg, draws 1.1 A at 120 V AC (60 Hz), has a nominal pumping speed of 1.1 $\text{m}^3 \text{hour}^{-1}$ at 60 Hz, and can achieve an ultimate pressure of 4 mbar (3 torr), or a factor of 6 better than required for the TPD 011 turbomolecular pump.

3.4 Software

The delivered instrument is operated by a program, LabMPA-final written in LabVIEW that controls a sampling valve to acquire the analyte, transfers the sample onto the gas chromatograph column, ramps the column temperature, measures the mass spectral ion intensities at a series of user-defined peaks, and records the data to disk in ASCII format.

Communication between LabMPA-final running on the laptop computer and the mass spectrometer system uses a PCMCIA plug-in card that contains a pair of RS-232 serial ports. This brings to three the total number of serial ports. The computer's built-in port (COM1) provides communication with the Ferran Scientific mass spectrometer's computer network interface (CNI). The two ports on the PCMCIA card (COM4 and COM5) address a pair of PID temperature controllers (Omega model CNI322-C24). One regulates the GC column temperature and the other reports the instantaneous temperature back to the laptop. The program also accesses a National Instruments DAQCard-DIO-24 PCMCIA plug-in card that uses 0 to 5 V logic signals to control the two valves and the column cooling fan.

Figure 8 shows the user interface that appears when the program is first opened. The major user controls appear on the left-hand side of the panel. At the top, masses to scan, is the list of ion masses that are acquired during each measurement. The next five items control the timing of the sampling and gc operation. The last of the group, set point, is the target maximum temperature achieved by the chromatograph. We identify this value as a "target" temperature, because the upper bound is

constrained by thermal loss to the surroundings and so is affected by ambient air temperature, humidity, and air currents.

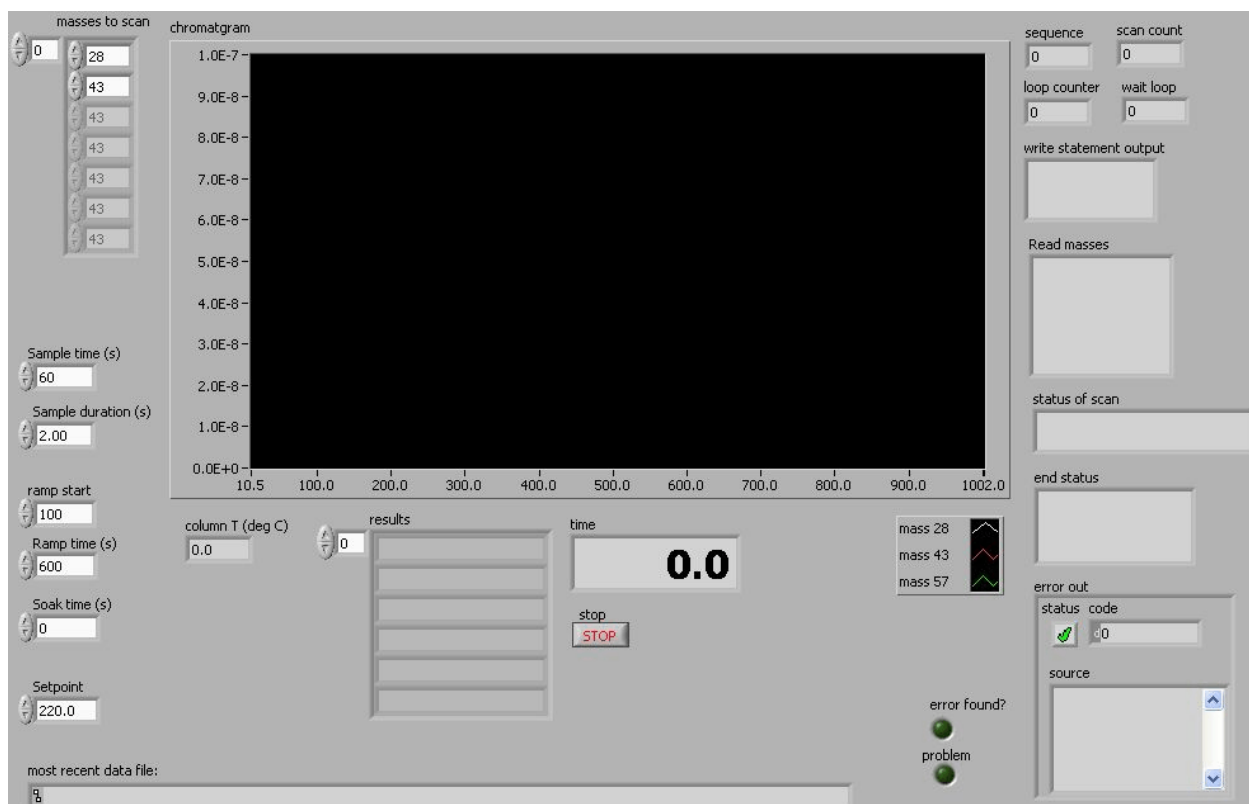


Figure 8 - Front panel of the LabVIEW program used to control the mass spectrometer system and collect and store the chromatographic data.

The central region of the panel, dominated by the chromatogram plot area, provides the measurement results as acquired including the actual column temperature, the most recently acquired ion signals (in torr partial pressure), and the elapsed time in seconds.

The right-hand region is used primarily for debugging and to help the user examine raw signals between the host computer and the Ferran controller.

Figure 9, shows the six major controls from the program main panel, and describes their use. This information is displayed graphically on a time line in Fig. 10. The setpoint temperature is a target value that, for

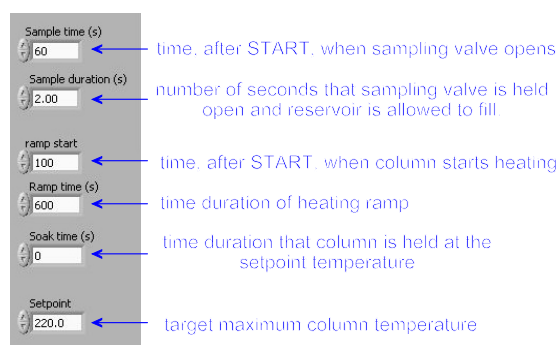


Figure 9 - Detail of program front panel showing the key timing and temperature controls.

temperatures ≥ 190 °C, may be limited by the ambient air temperature, humidity, and air currents. We have found it useful to set a target above the maximum achievable temperature since this drives the PID temperature controller more strongly at the upper end of the temperature cycle and helps maintain a linear temperature ramp.

Experimental results are written to disk in ASCII format comma-delineated rows and columns. In all cases, the first column is the measurement time (relative to 0.0 at the start of the run), followed by the column temperature in °C. Then comes the

partial pressures in torr for the selected ion signals. The temporal resolution is determined by the number of mass peaks measured because the Ferran computer network interface establishes a minimum dwell time of 0.6 seconds per mass peak. The text box below is a portion of a data set acquired when the instrument was being set up and demonstrated at NASA Glenn Research Center in August 2004. The demonstration used a calibration mixture of alkanes (1000 ppm each of methane, ethane, propane, *n*-butane, *n*-pentane, and *n*-hexane).

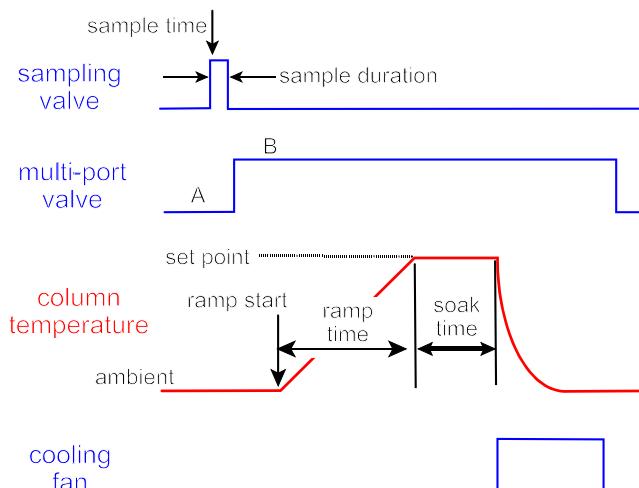


Figure 10 - Timing diagram for the LabVIEW program.

```
time, temp, amu0028, amu0043, amu0057
1.0586E+1, 2.6600E+1, 8.3660E-6, 7.6010E-7, 6.3700E-7
1.3979E+1, 2.6600E+1, 6.8930E-6, 7.5580E-7, 6.1260E-7
1.8120E+1, 2.6500E+1, 6.4640E-6, 7.4810E-7, 6.3390E-7
2.2302E+1, 2.6500E+1, 6.1880E-6, 7.4990E-7, 6.2860E-7
2.6548E+1, 2.6500E+1, 6.2140E-6, 7.4610E-7, 6.2650E-7
3.0242E+1, 2.6500E+1, 5.9070E-6, 7.4630E-7, 6.2370E-7
3.4475E+1, 2.6600E+1, 6.0410E-6, 7.2740E-7, 6.3020E-7
3.8620E+1, 2.6600E+1, 5.8840E-6, 7.4380E-7, 6.3960E-7
```

The full data set is shown graphically in Fig. 11. The large mass 28 background and the noise are due to some air contaminating the helium carrier gas.

The LabVIEW program LabMPA-final automatically saves each chromatogram to disk using an automatically generated filename AUTOSAVE_nnnnn.TXT where nnnnn is a number beginning with 00000 that is incremented to prevent duplicating files. The user is given the opportunity to rename the autosaved file at the end of each run.

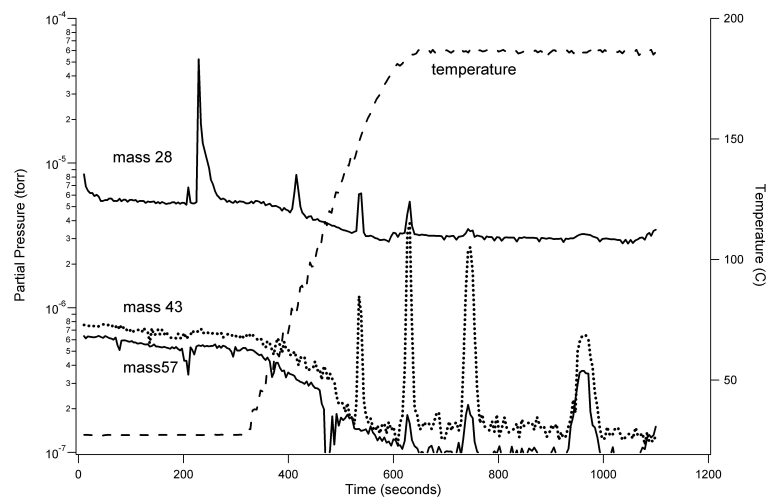


Figure 11 - Chromatographic and temperature data from demonstration run.

Appendix A

LabVIEW source code

LabMPA-final.vi

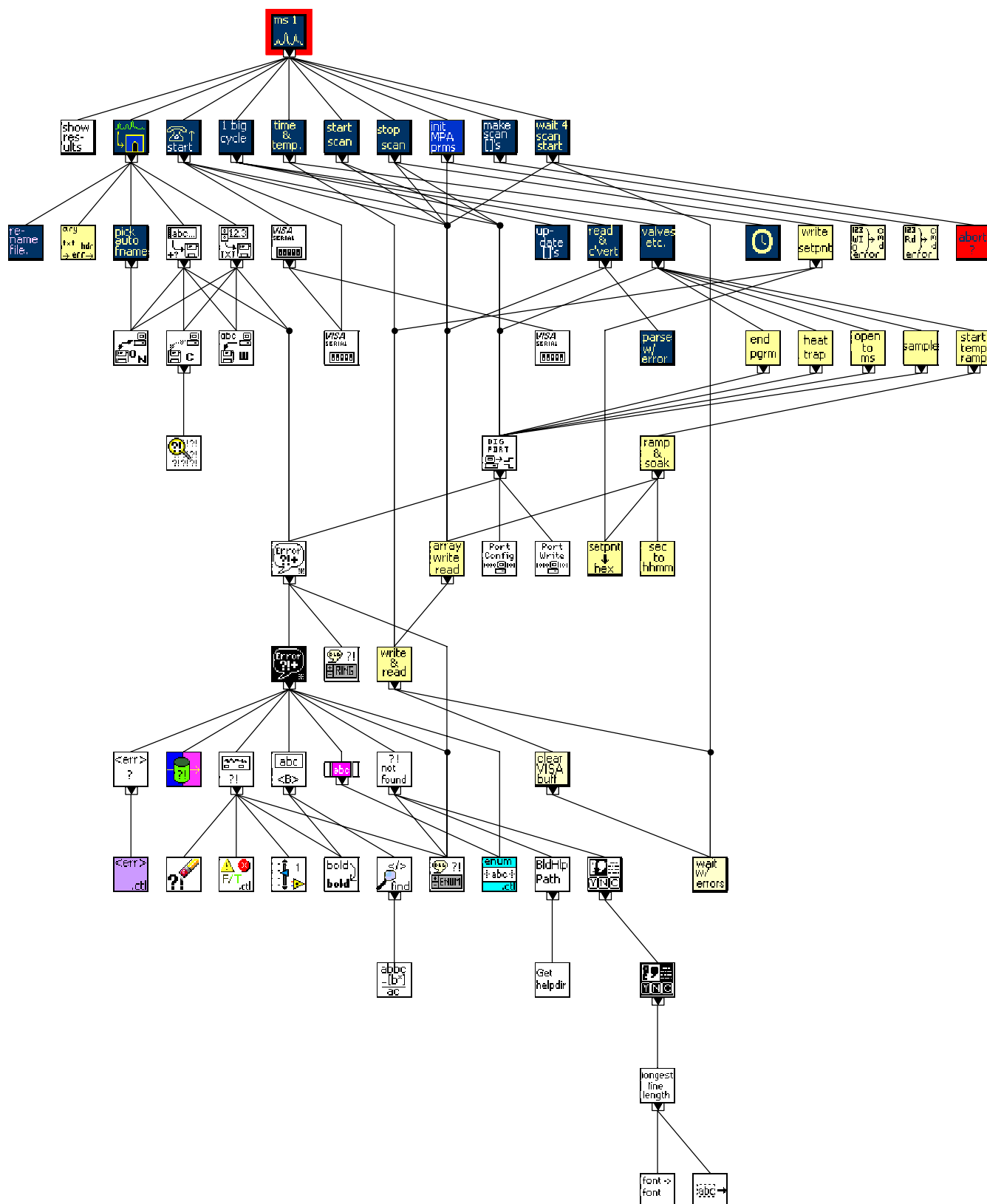
C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA.IIb\ITD\
LabMPA-final.vi

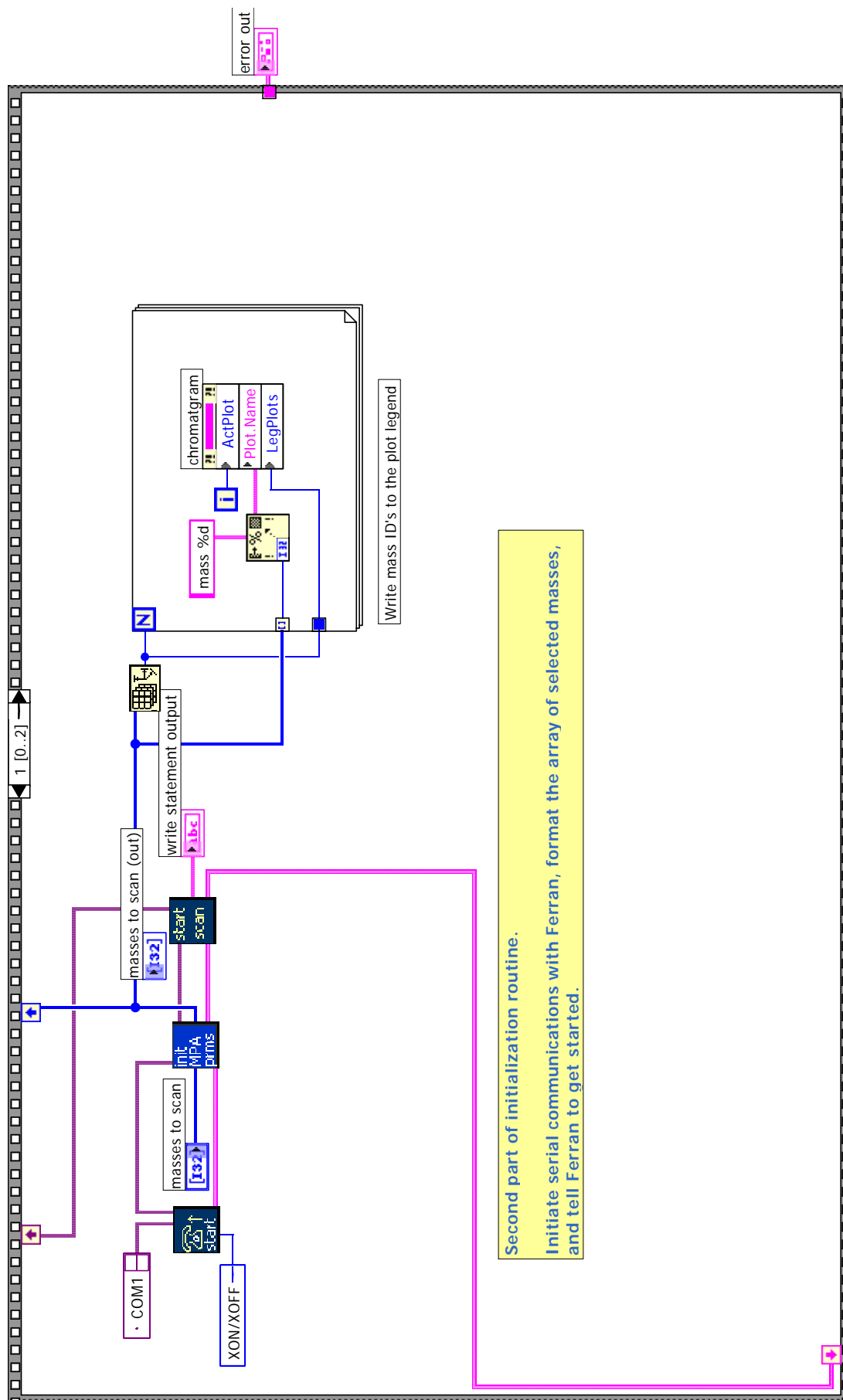
Last modified on 8/31/2004 at 2:08 PM

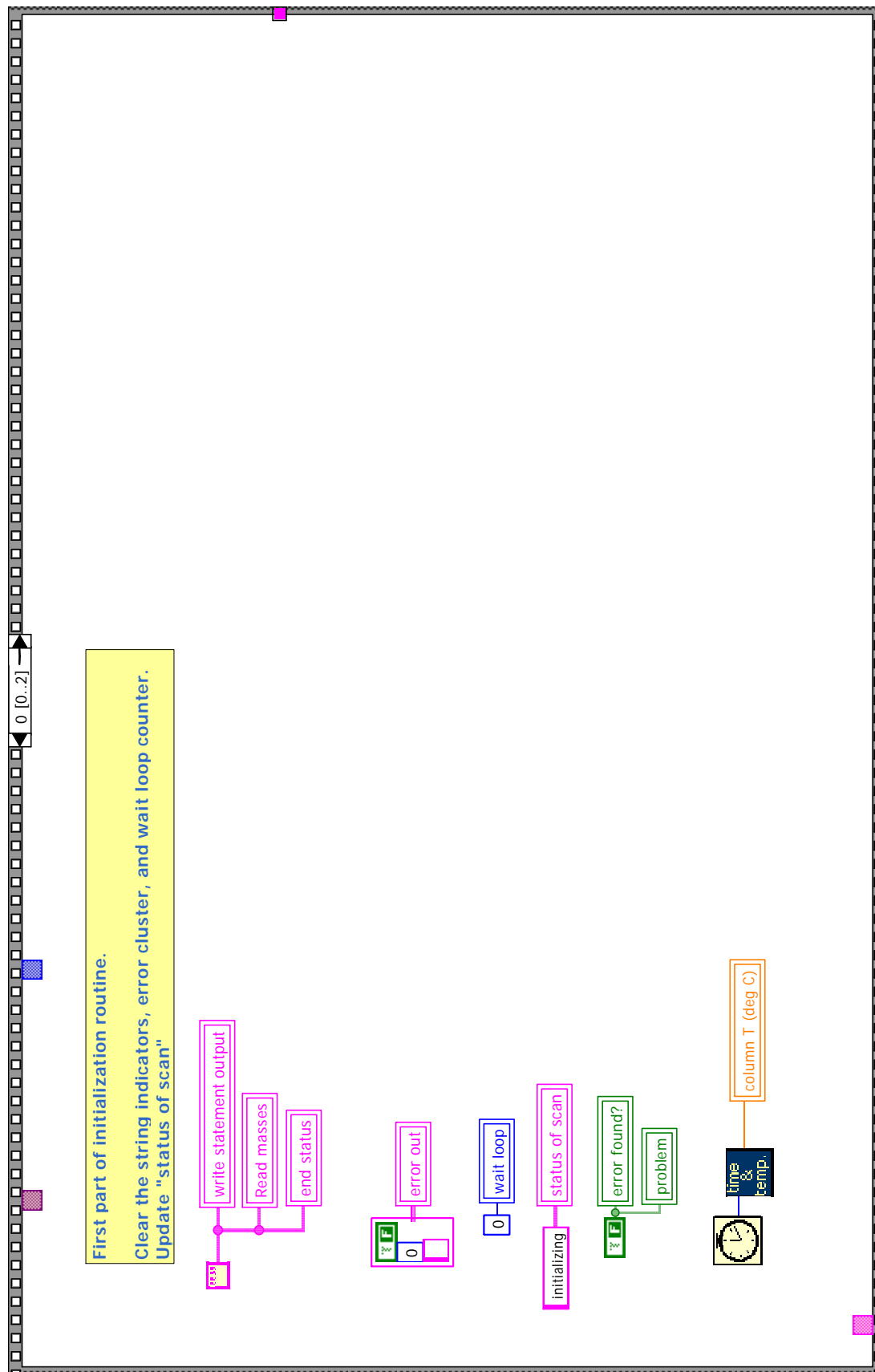
Printed on 12/20/2004 at 4:14 PM



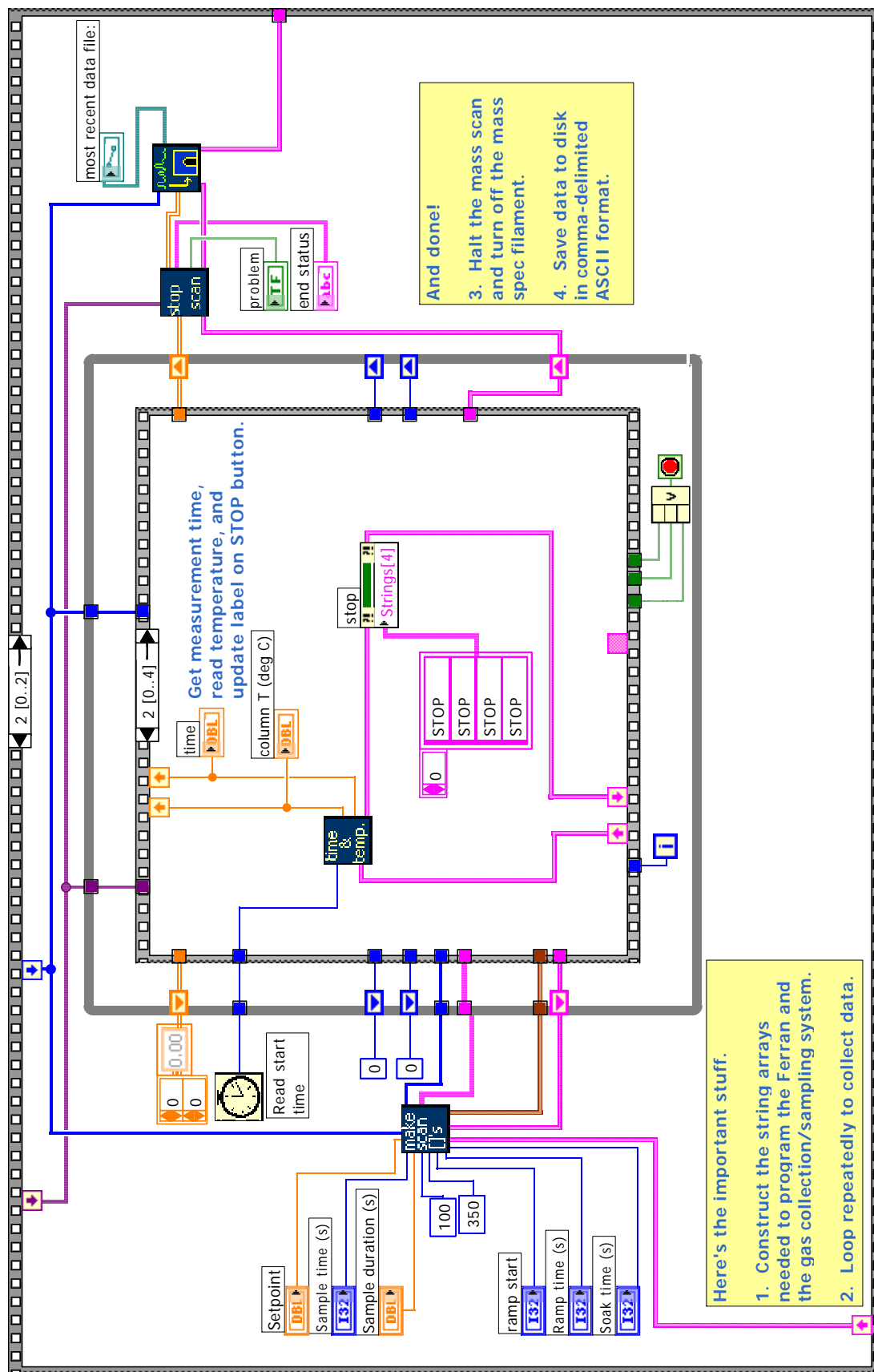
Printed on 12/20/2004 at 4:16 PM

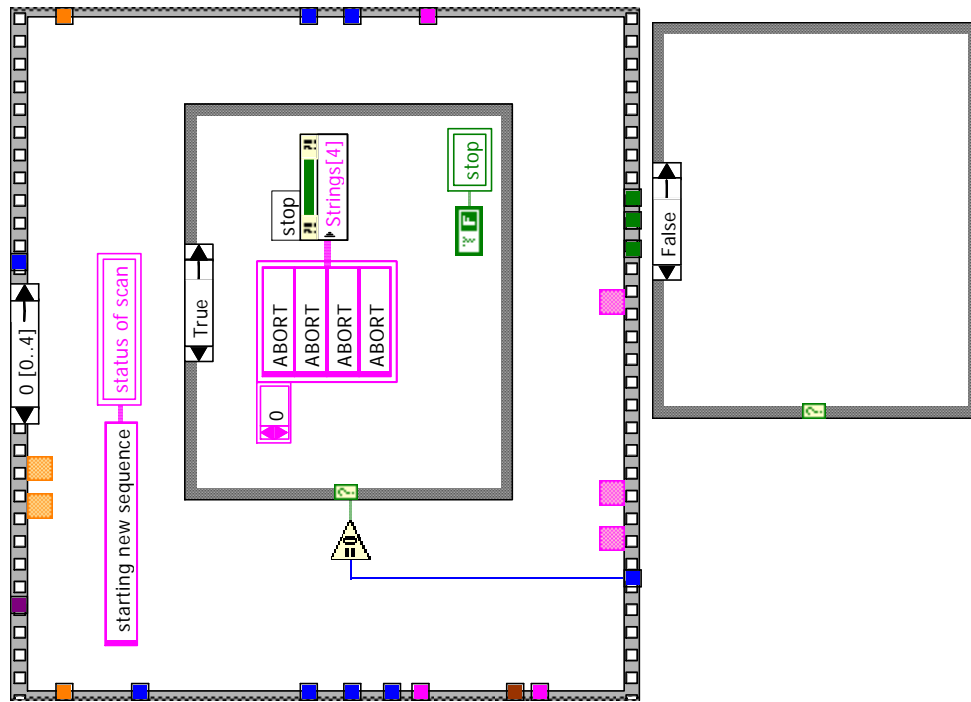


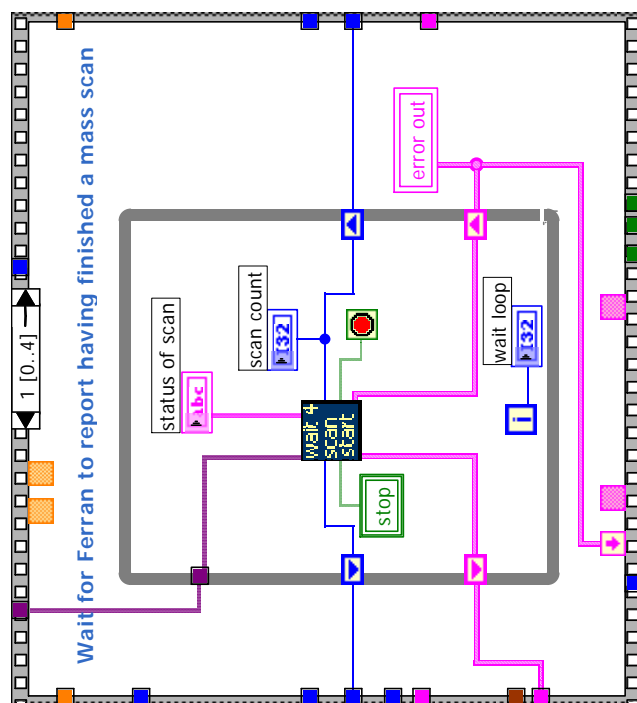


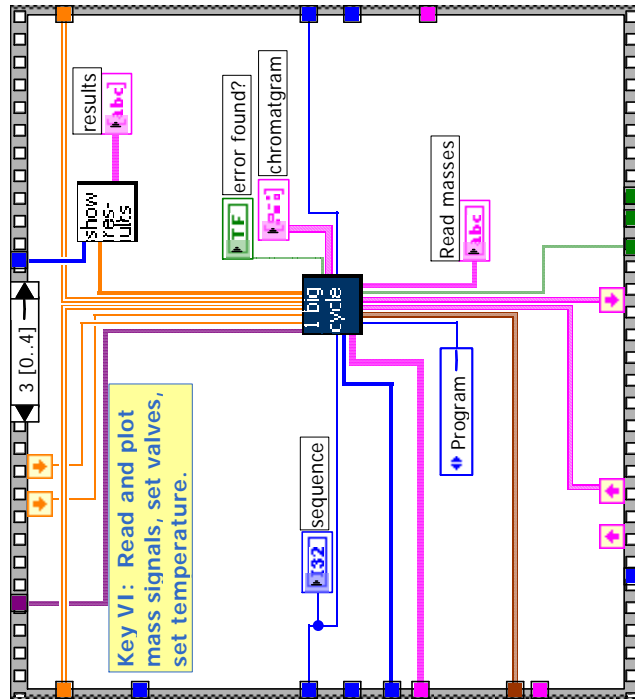


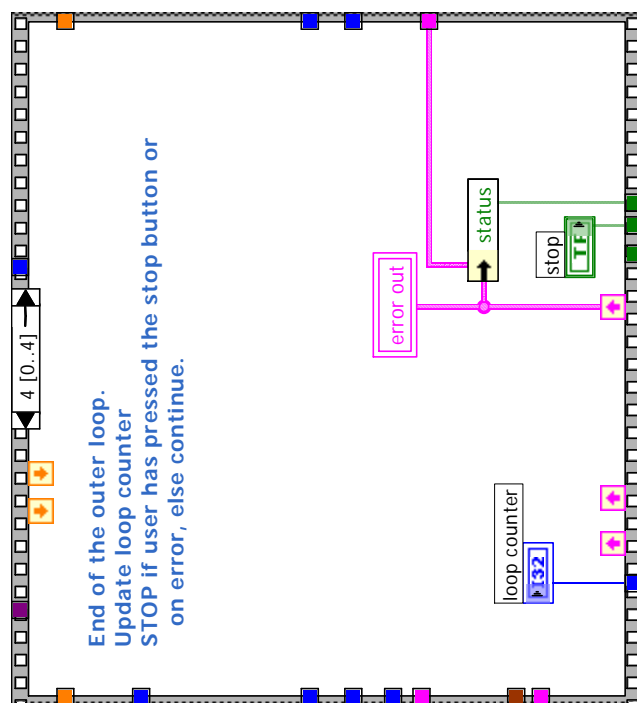
LabMPA-final.vi
 C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA.1lb\ITD\LabMPA-final.vi
 Last modified on 8/31/2004 at 2:08 PM
 Printed on 12/20/2004 at 4:18 PM



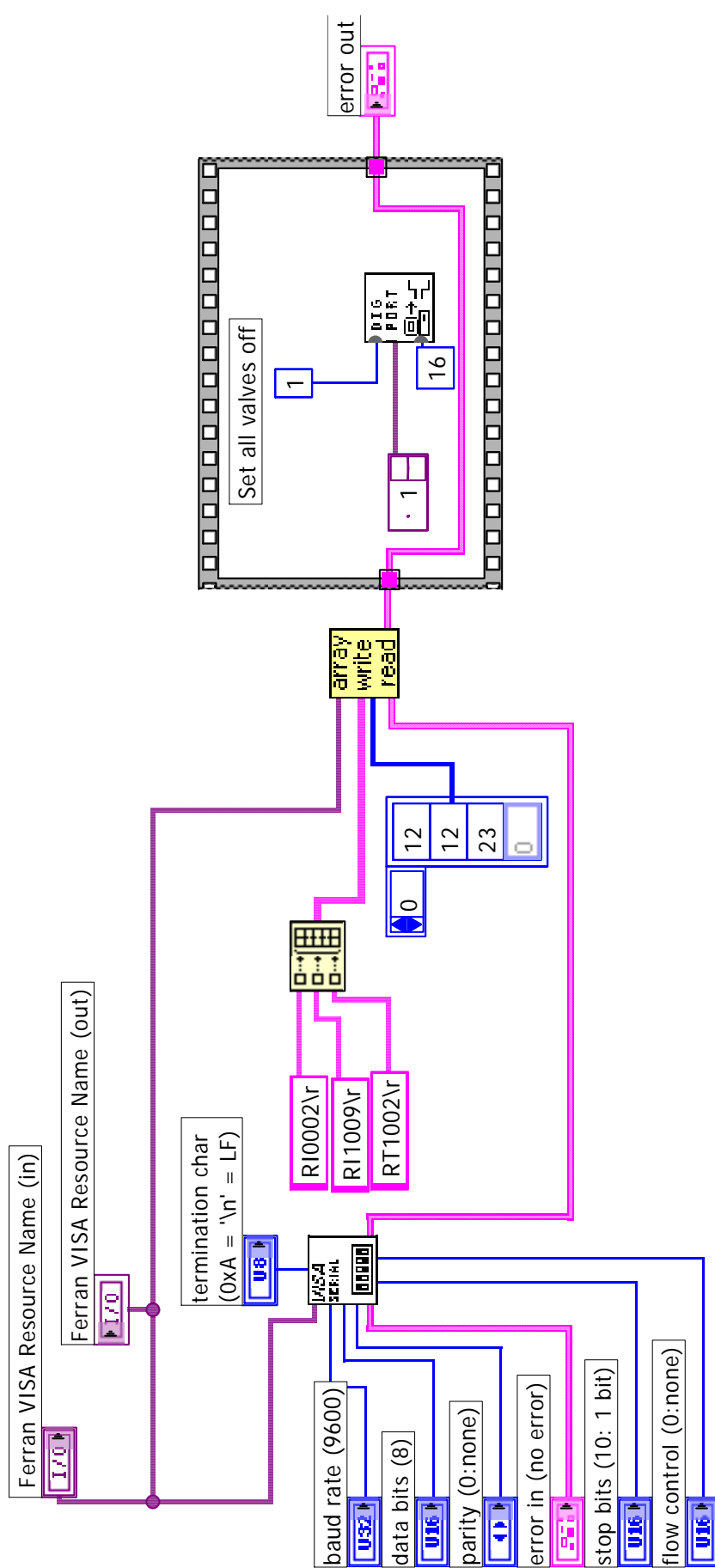






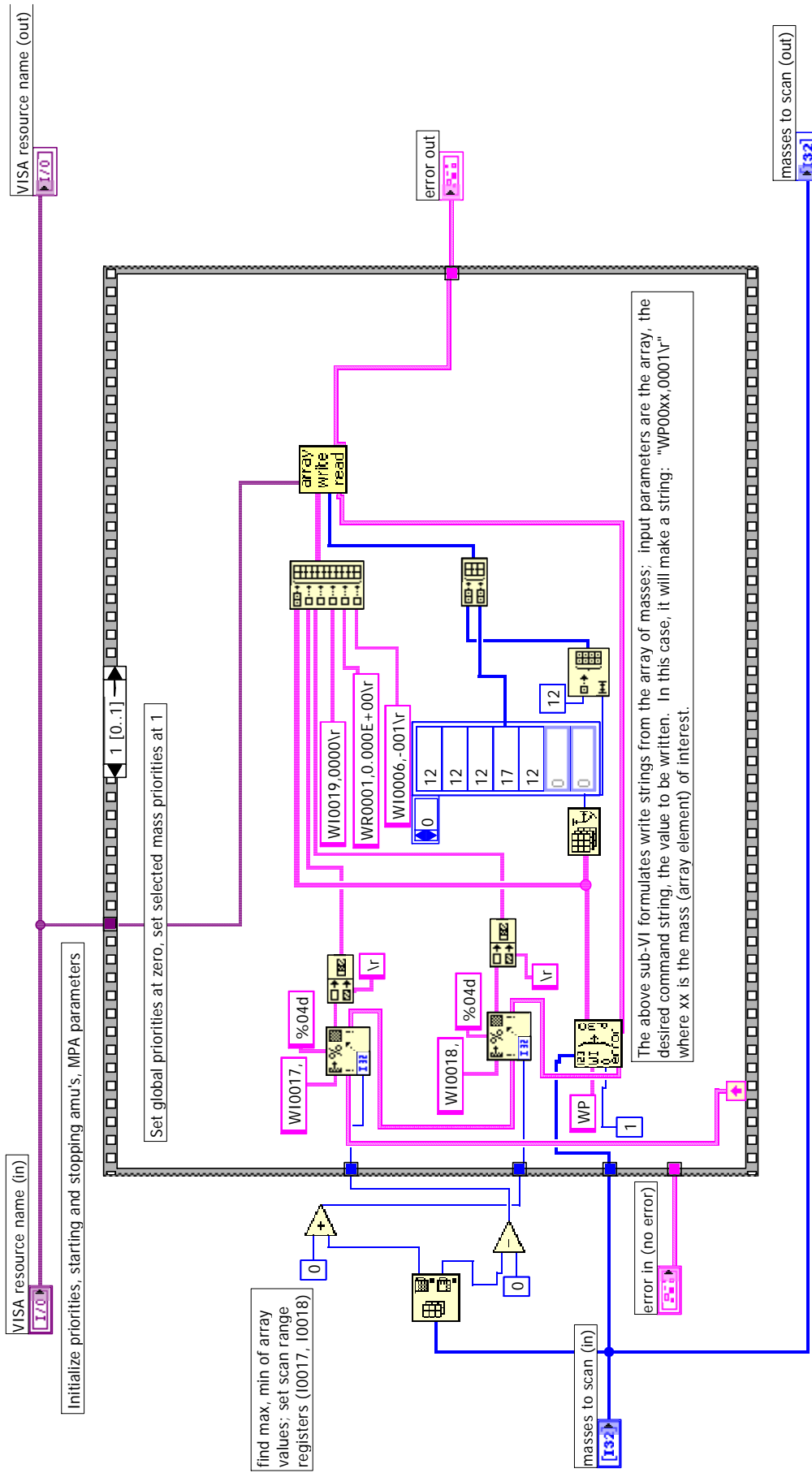


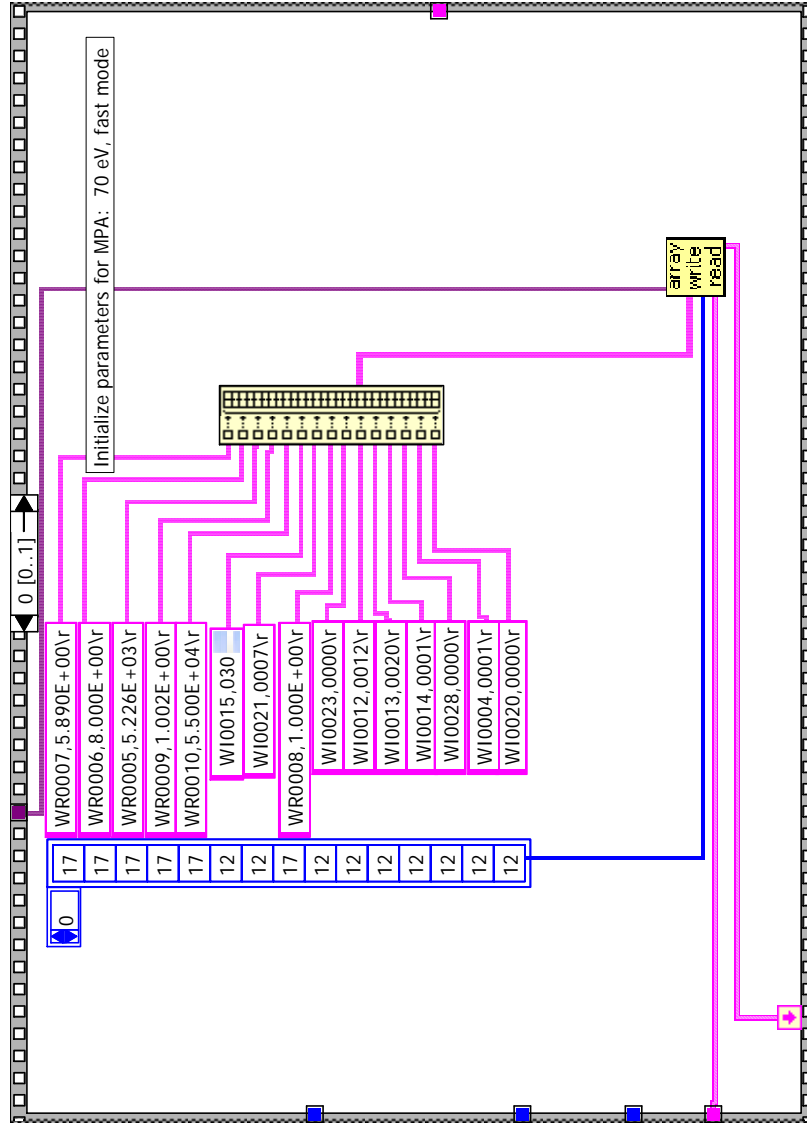
DSB init communications and close all valves.vi
C:\WPDocs\Contracts\C01-07 (NASA ITD mass
Last modified on 8/31/2004 at 2:08 PM
Printed on 12/20/2004 at 4:20 PM



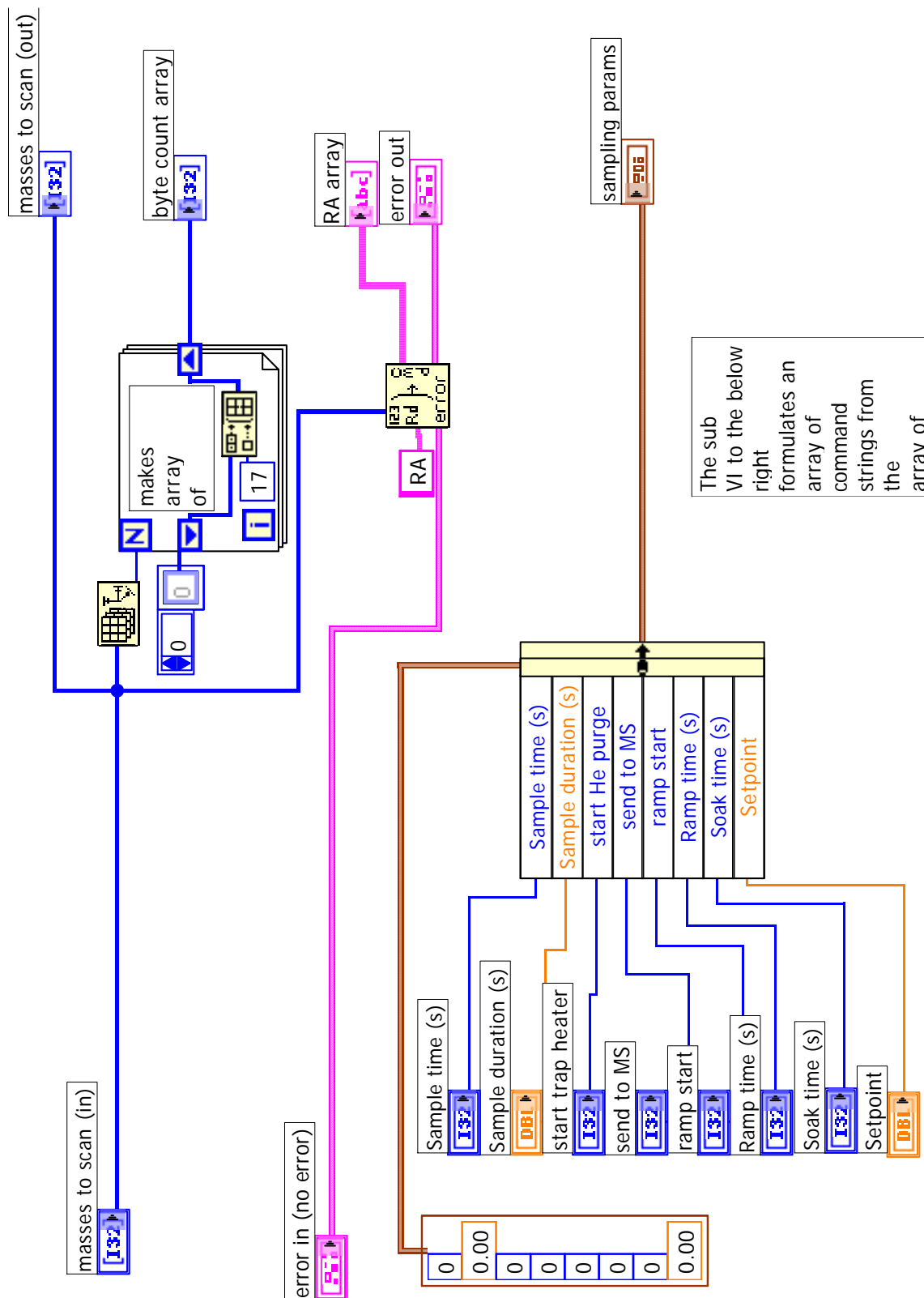
DSB init priorities, start stop amus, MPA params.vi
C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA.11b\DSB init priorities, start stop amus, MPA
params.vi

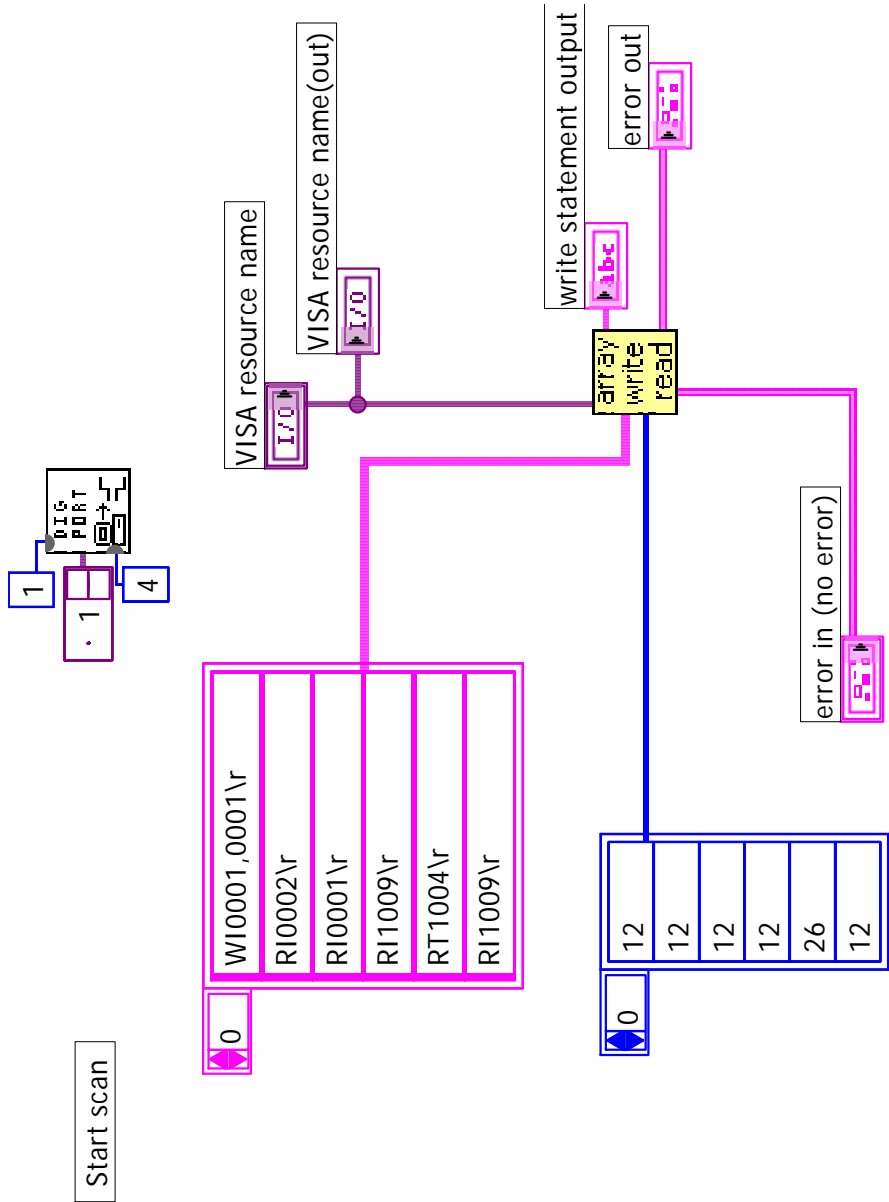
Last modified on 8/31/2004 at 2:08 PM
Printed on 12/20/2004 at 4:21 PM

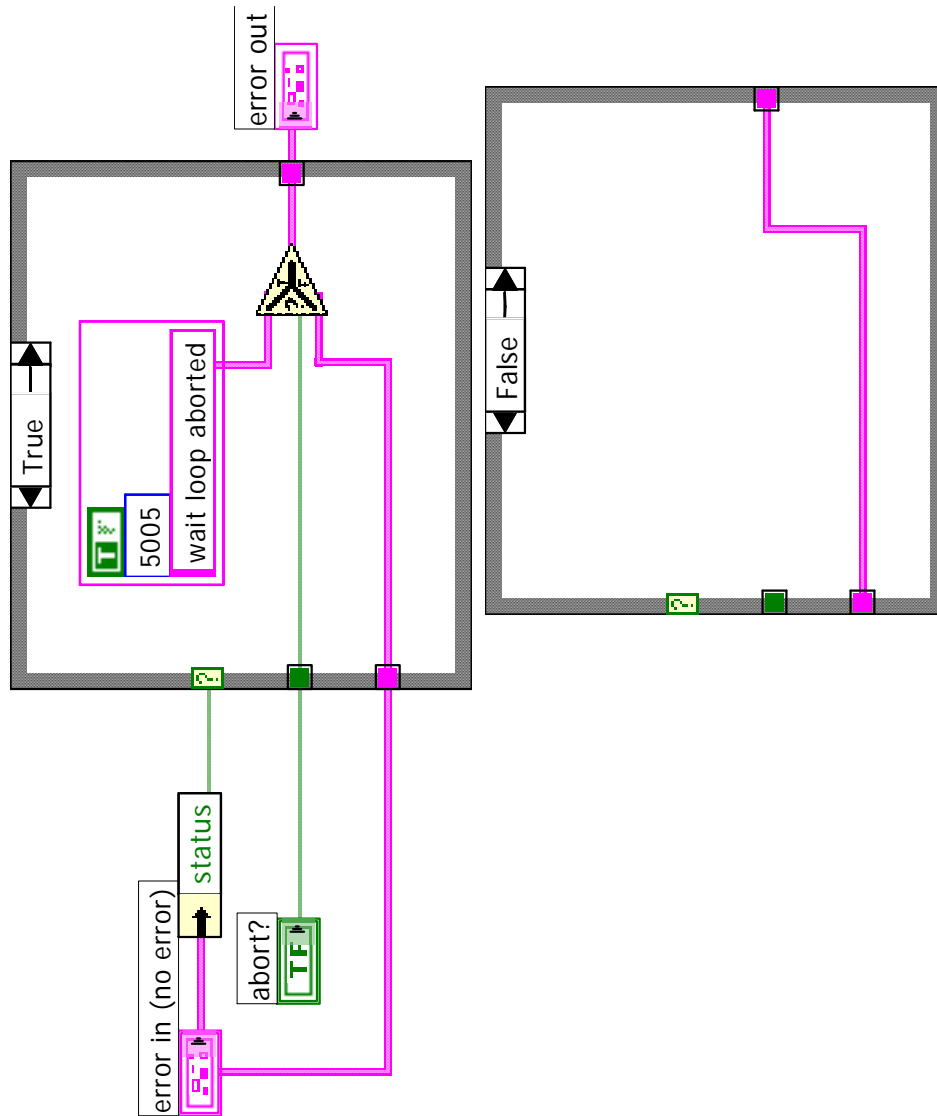




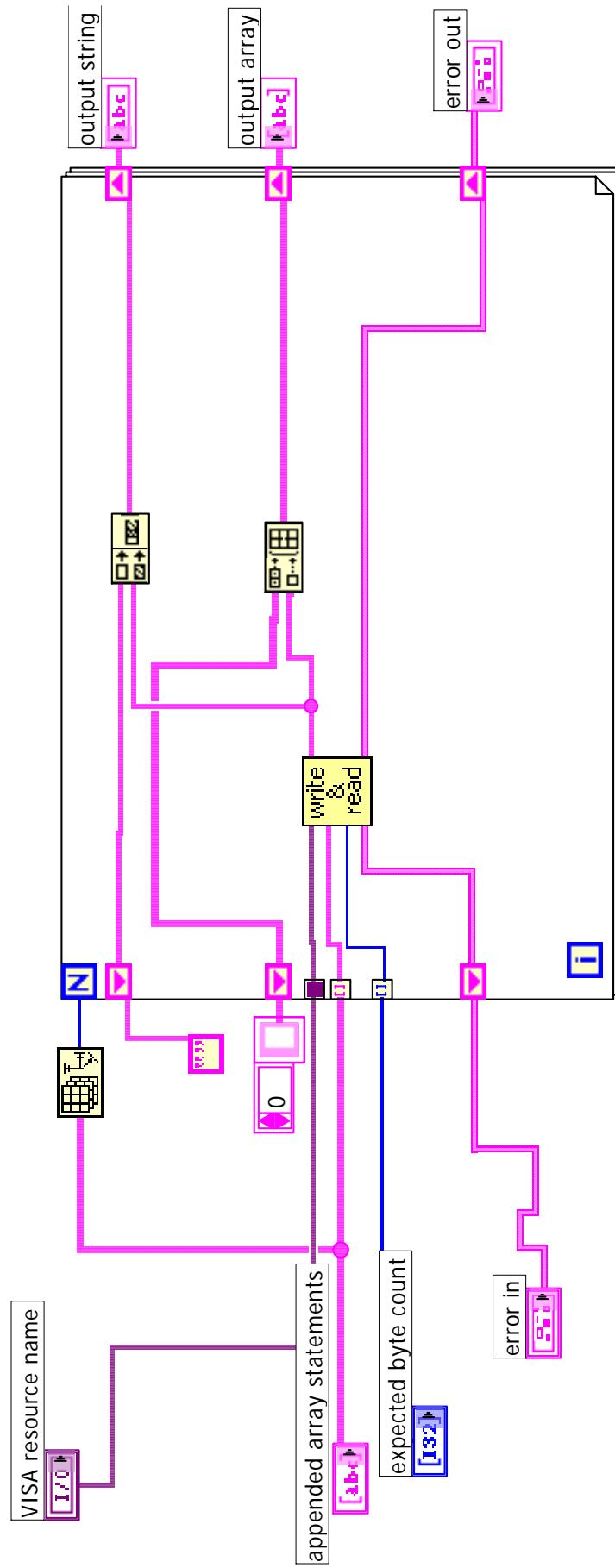
DSB set up scan arrays and cluster.vi
 C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA.1lb\DSB set up scan arrays and cluster.vi
 Last modified on 8/31/2004 at 2:08 PM
 Printed on 12/20/2004 at 4:23 PM



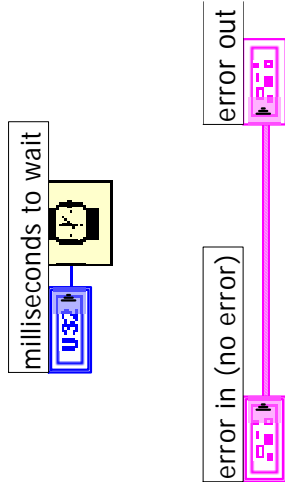


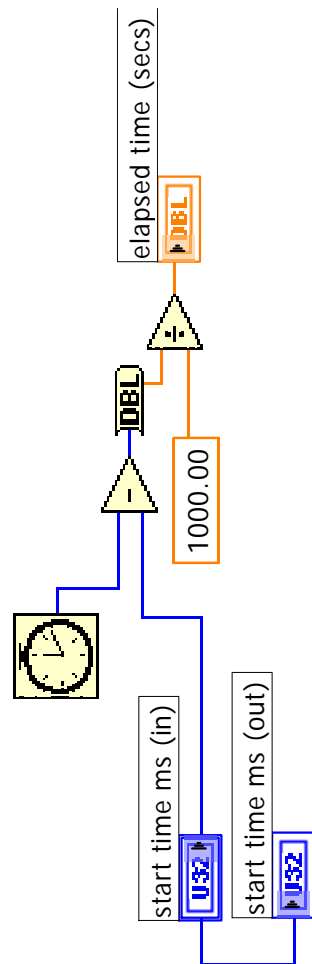


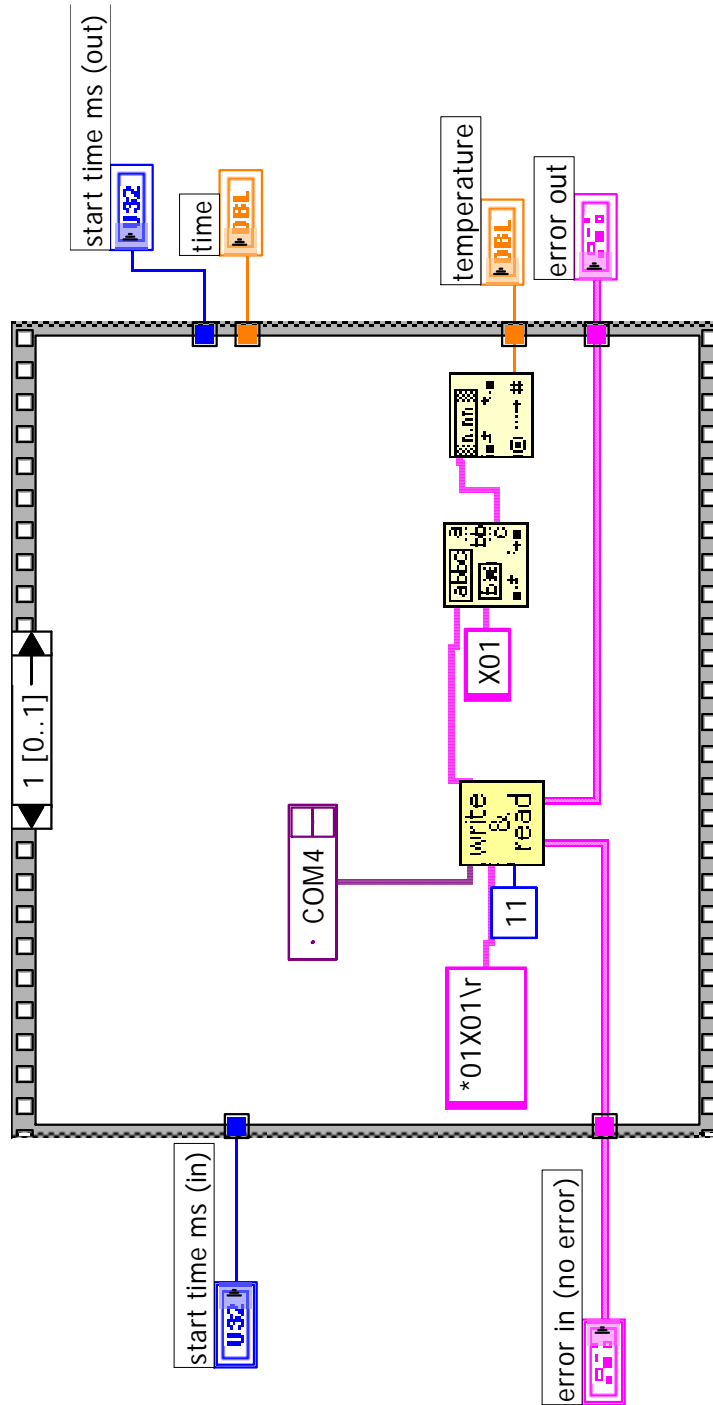
Initialize VISA: Inputs an appended array of string expressions to write and outputs the values. The number of bytes initially at the serial port is set to zero.



wait with error clusters.vi
C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA.1lb\wait with error clusters.vi
Last modified on 8/31/2004 at 2:08 PM
Printed on 12/20/2004 at 4:28 PM







Use sequence structure to guarantee that time is measured first then temperature.

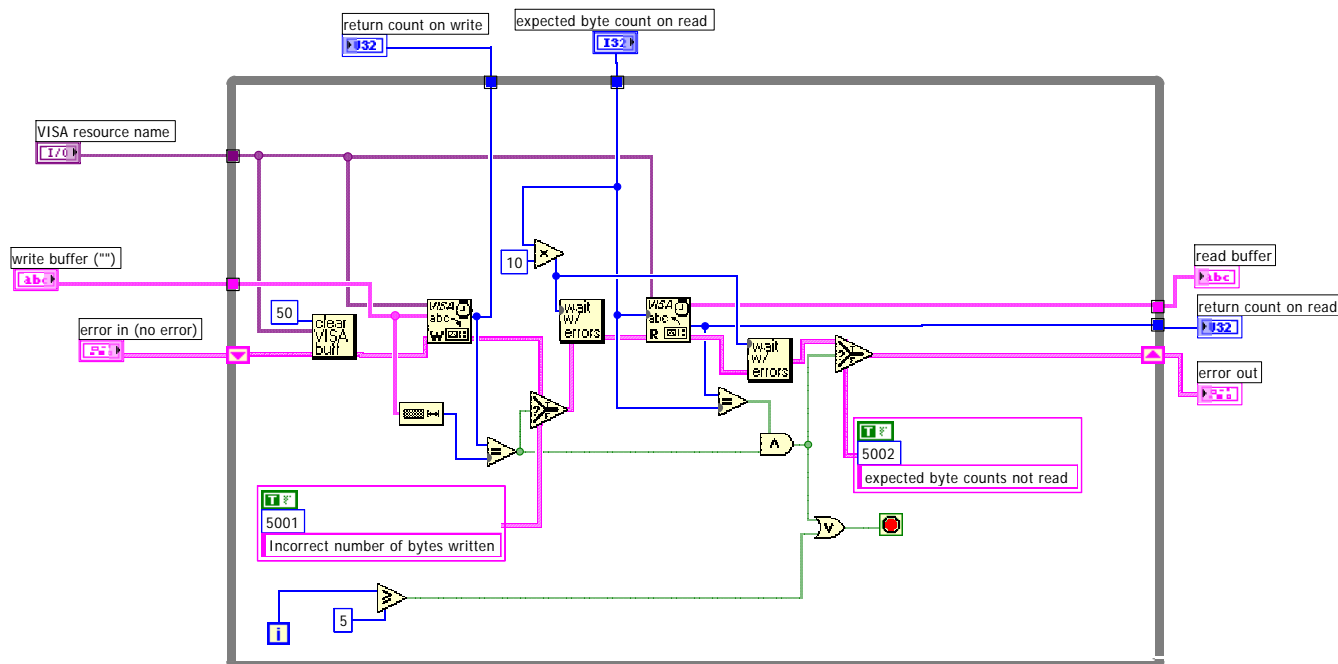
VISAwriteReadMAZ.vi

C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA.Ilb\

VISAwriteReadMAZ.vi

Last modified on 8/31/2004 at 2:08 PM

Printed on 12/20/2004 at 4:31 PM



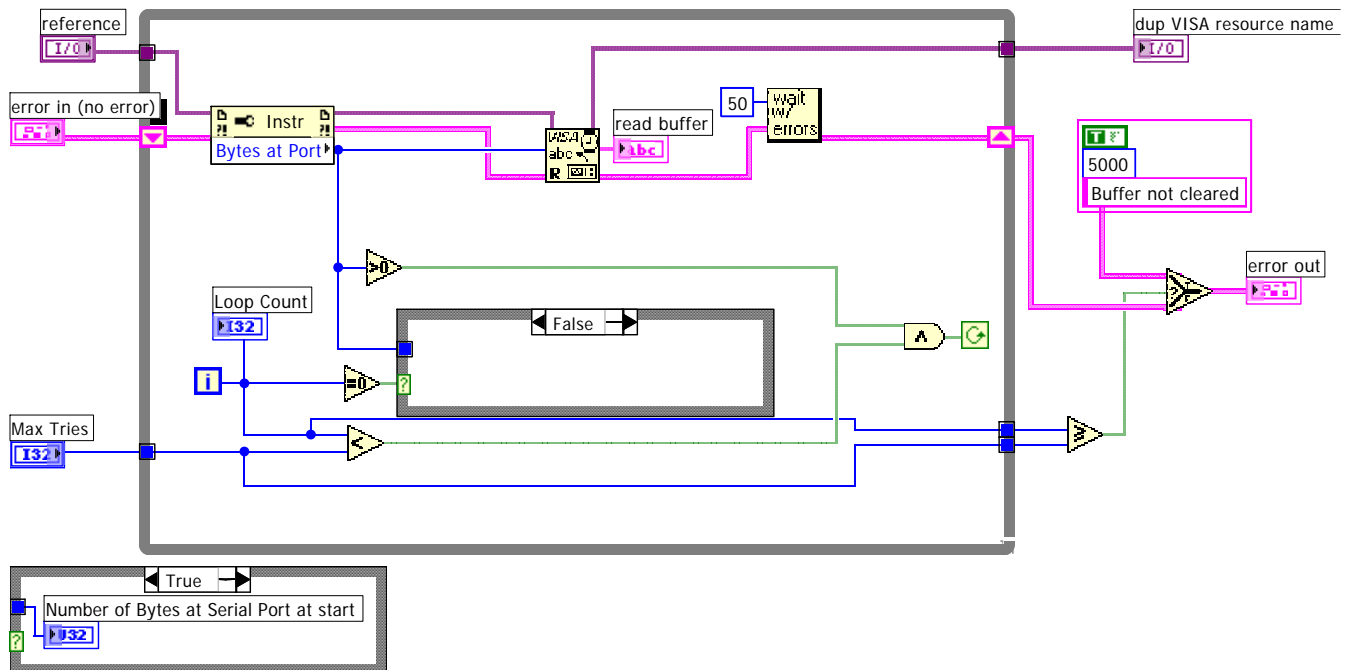


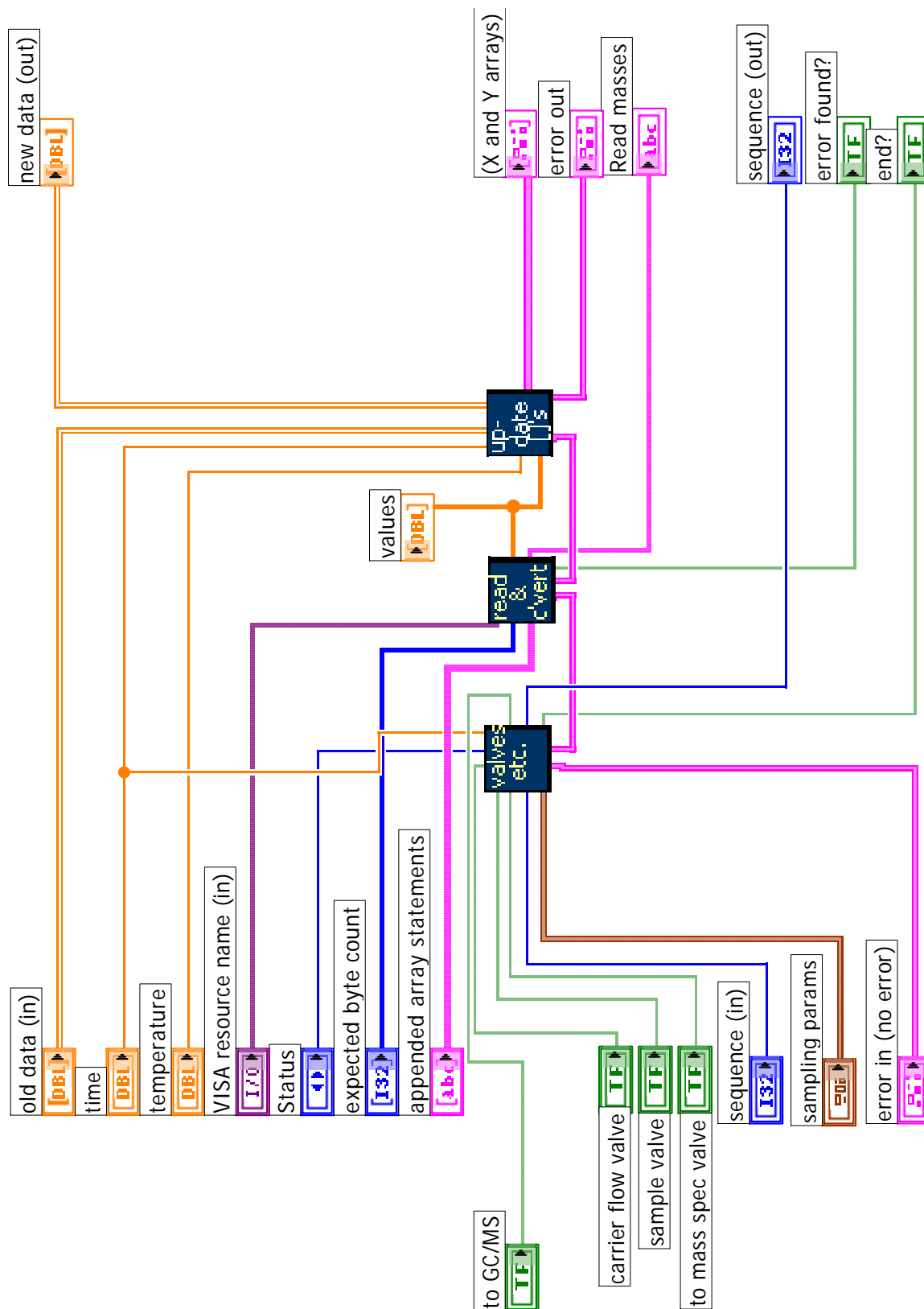
DSB clear buffer.vi

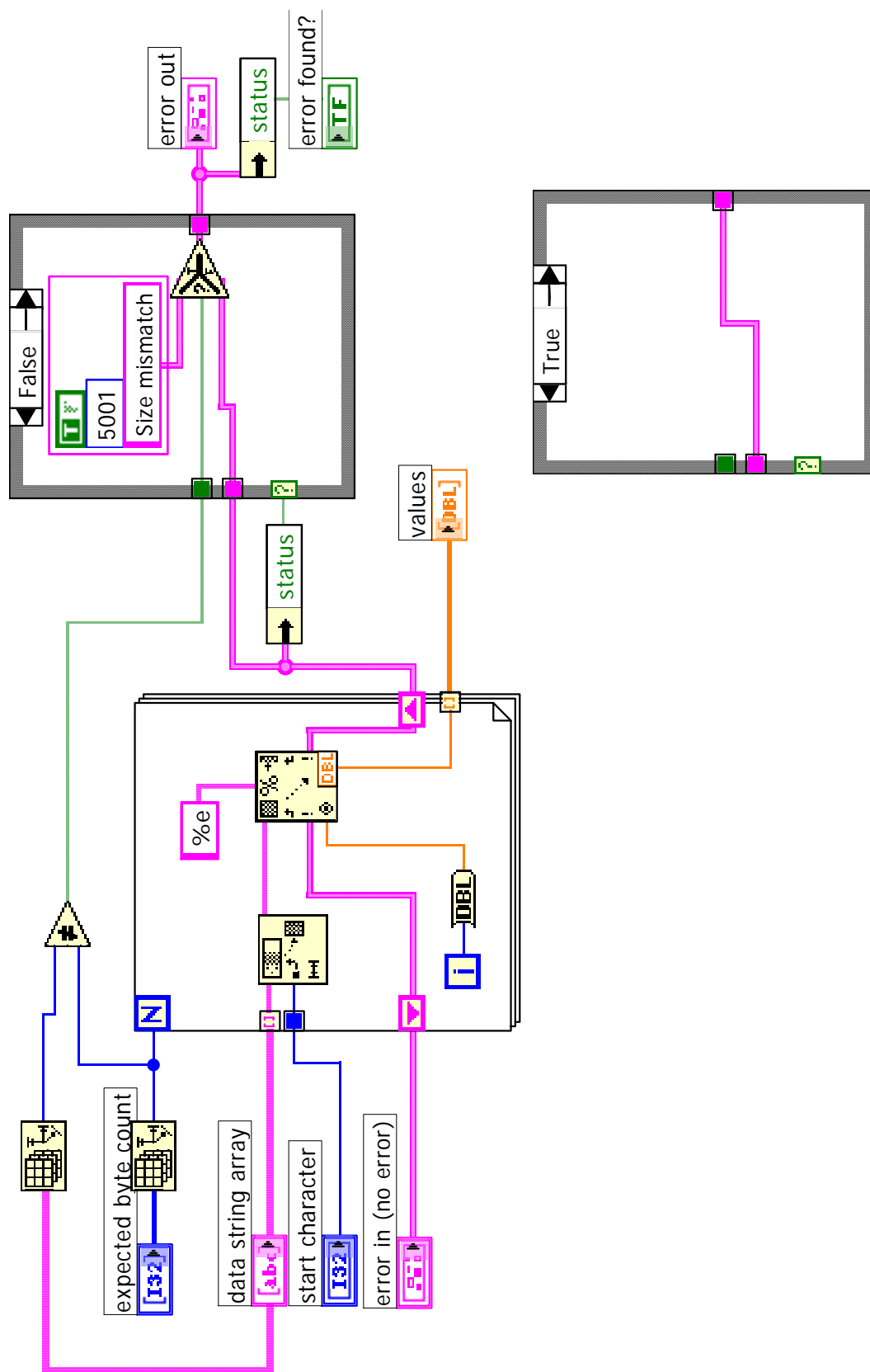
C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA.IIb\DSB clear buffer.vi

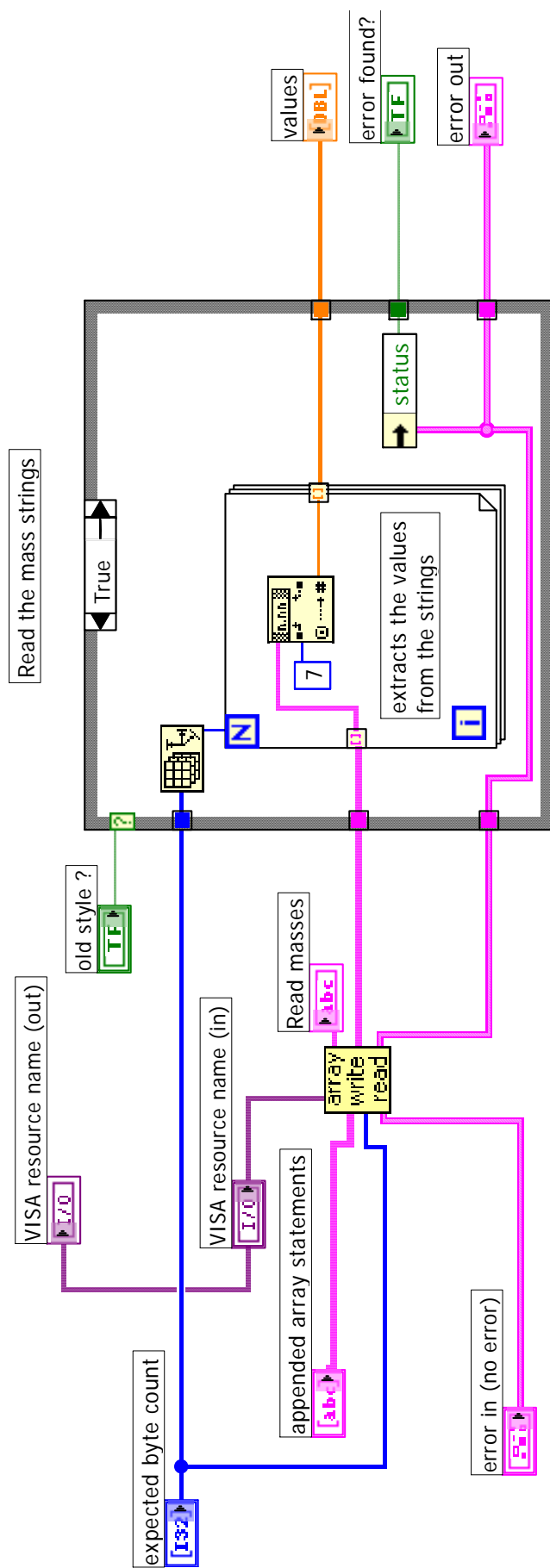
Last modified on 8/31/2004 at 2:08 PM

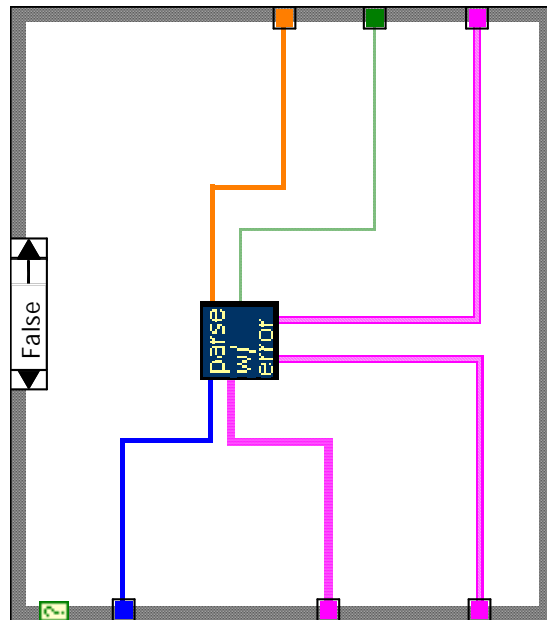
Printed on 12/20/2004 at 4:32 PM



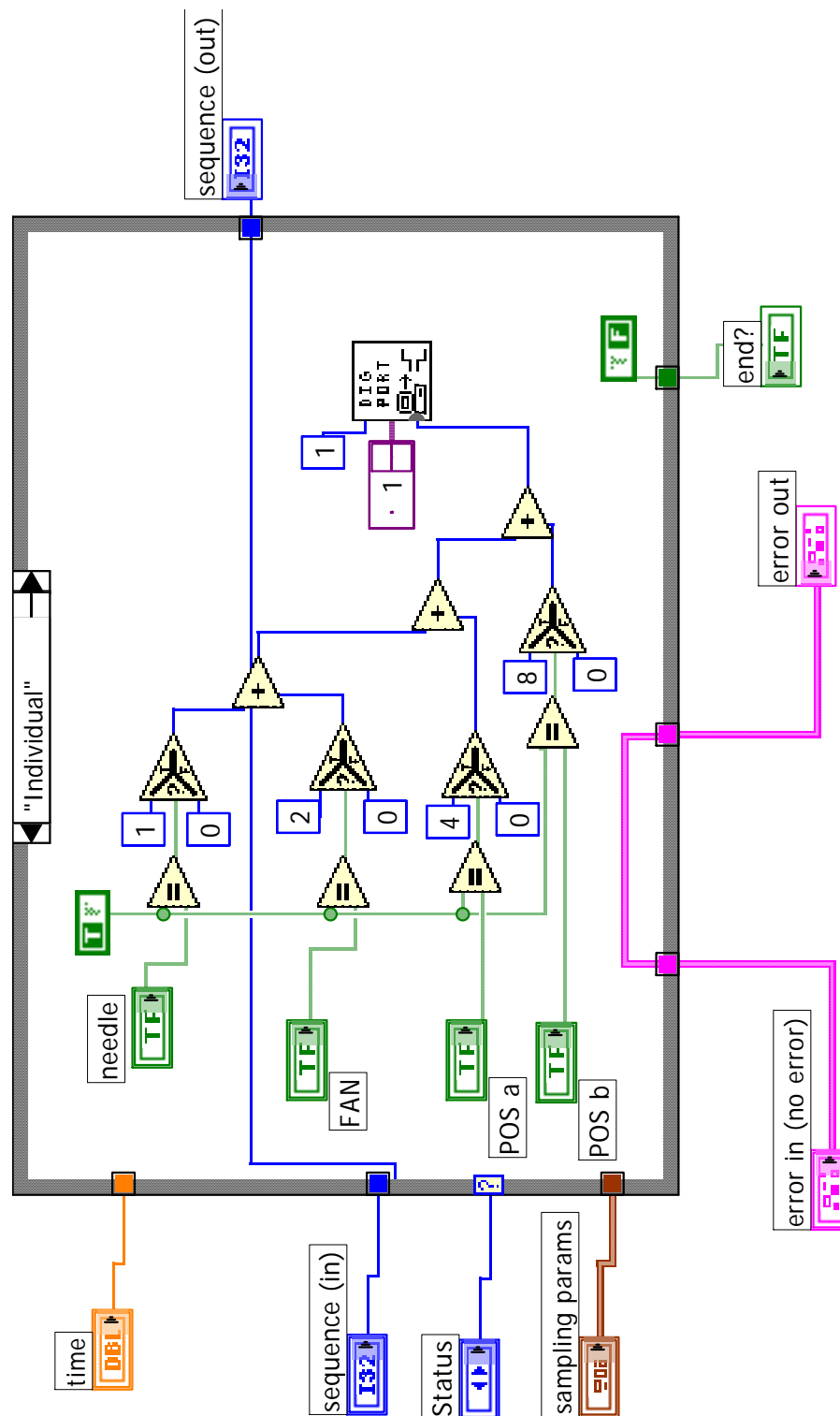


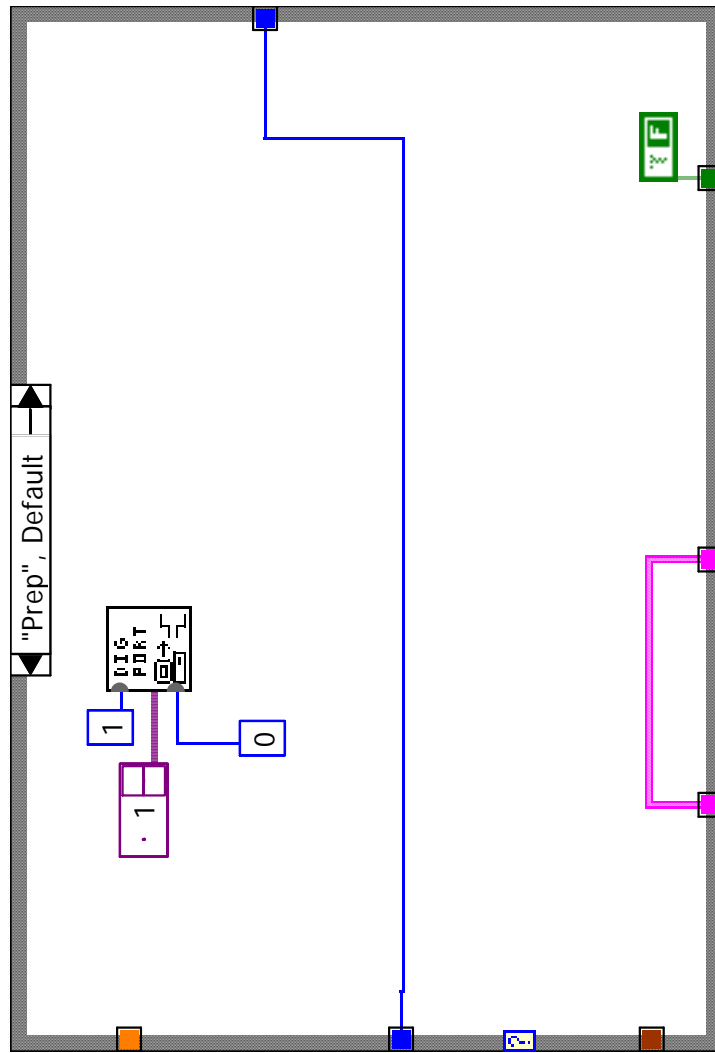


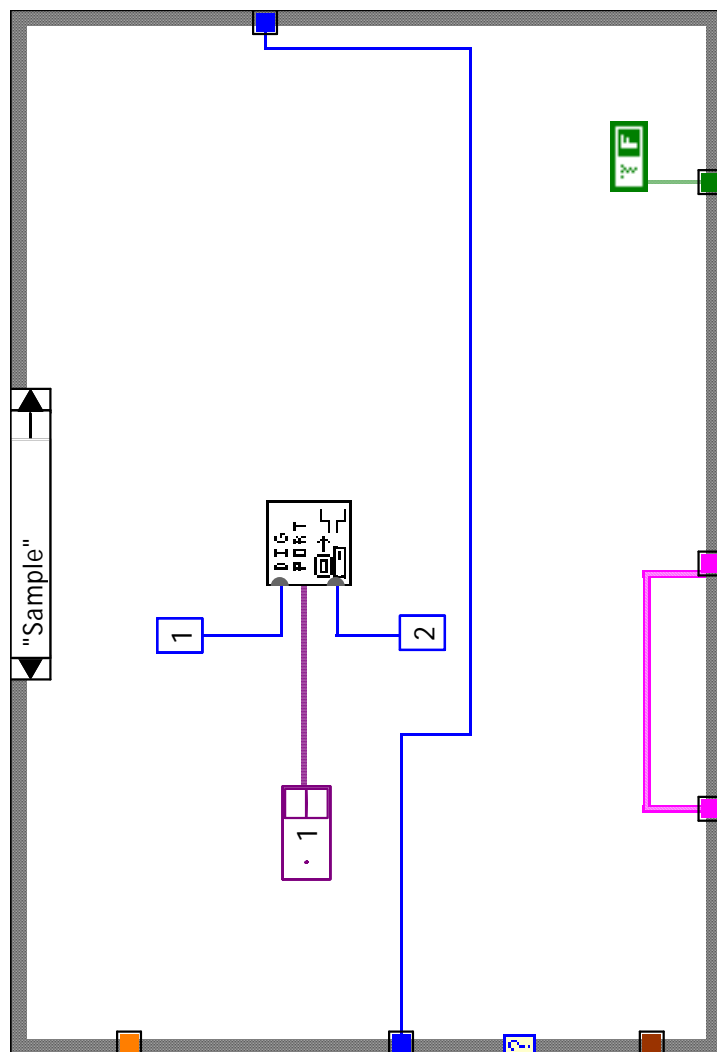




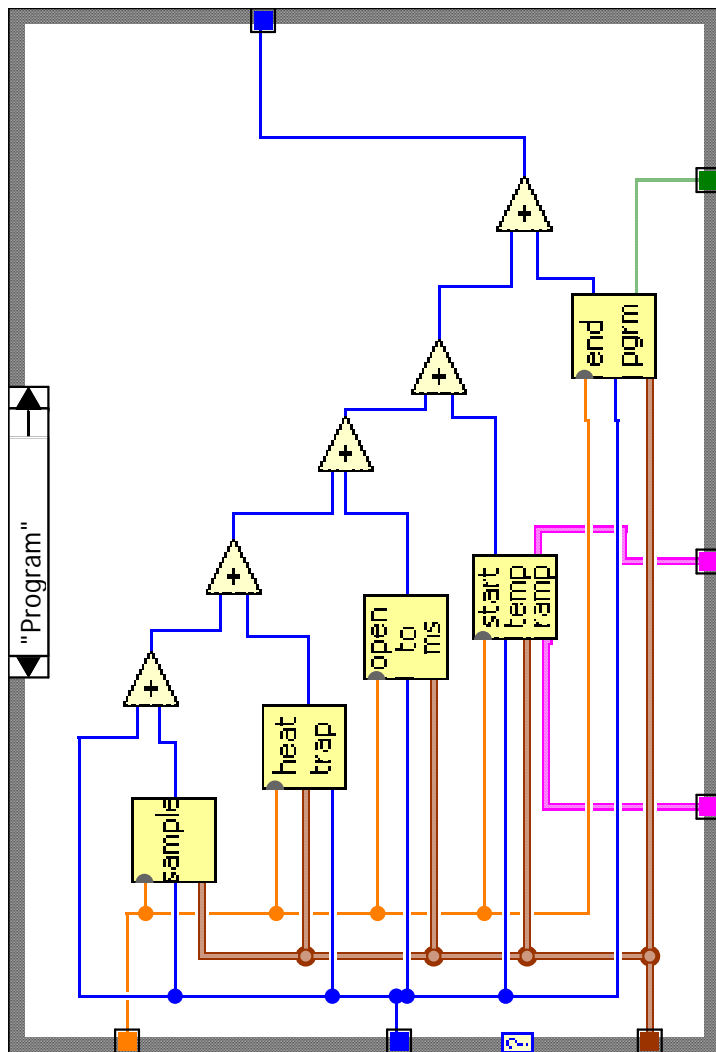
DSB valve controls, etc.vi
 C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA.Ilb\DSB valve controls, etc.vi
 Last modified on 8/31/2004 at 2:08 PM
 Printed on 12/20/2004 at 4:37 PM

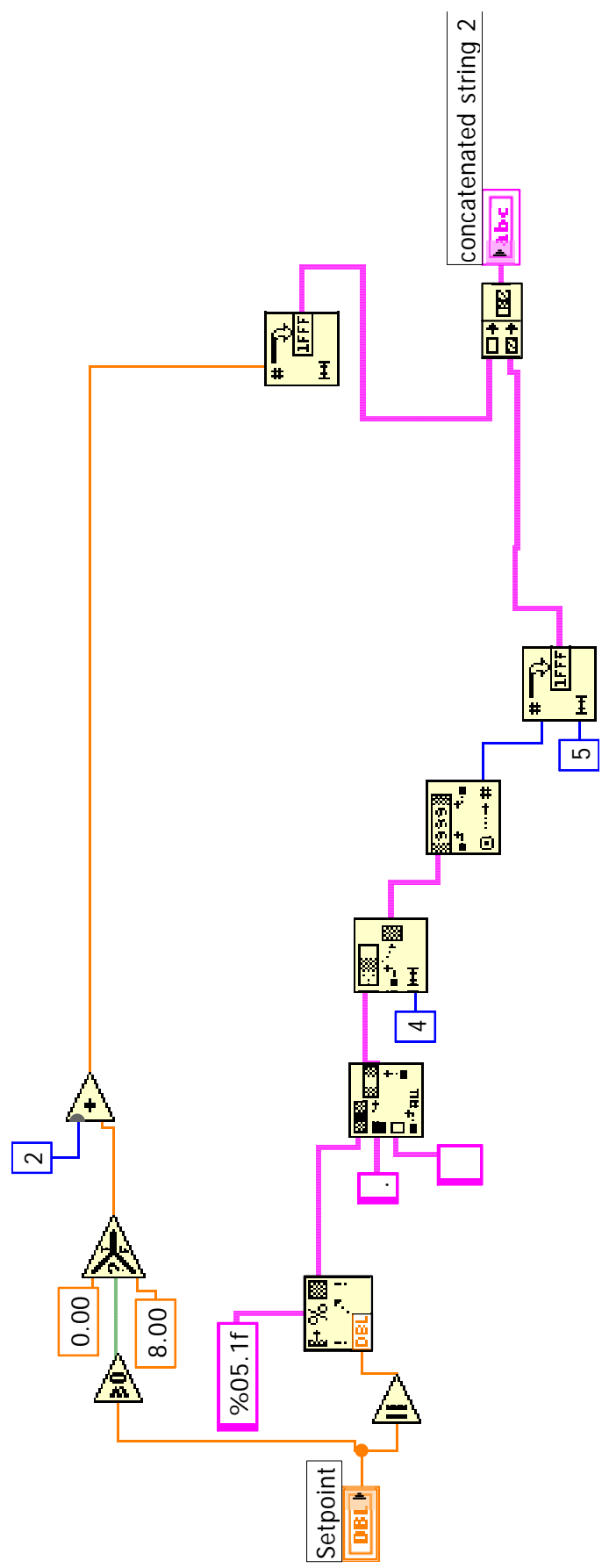


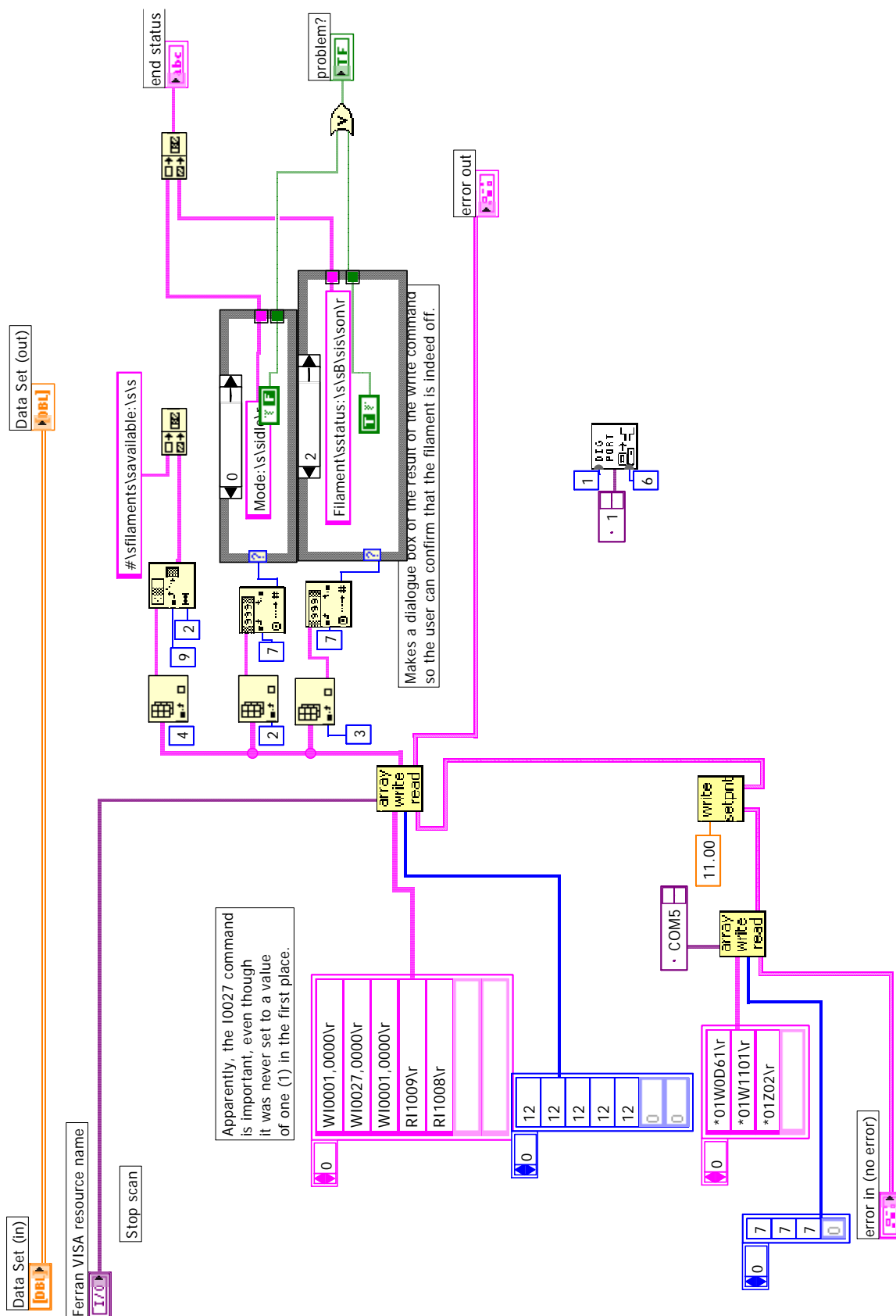


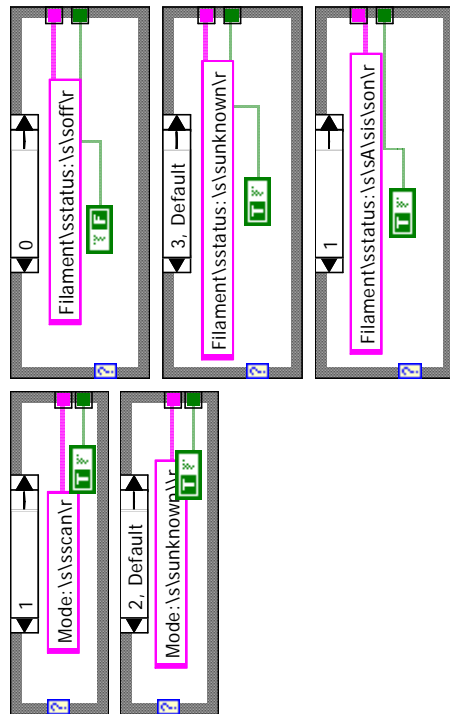


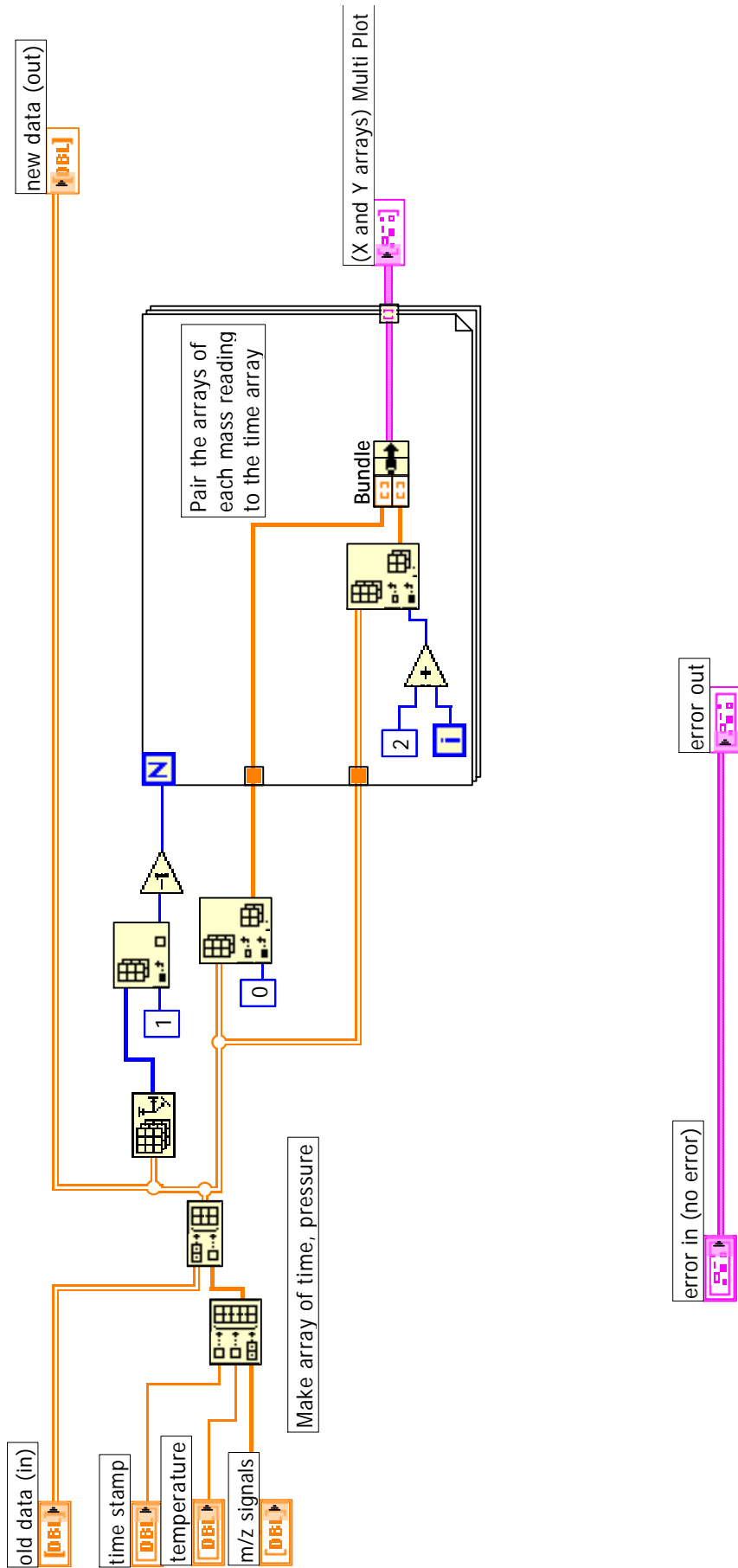
DSB valve controls, etc.vi
C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA_11b\DSB valve controls, etc.vi
Last modified on 8/31/2004 at 2:08 PM
Printed on 12/20/2004 at 4:37 PM

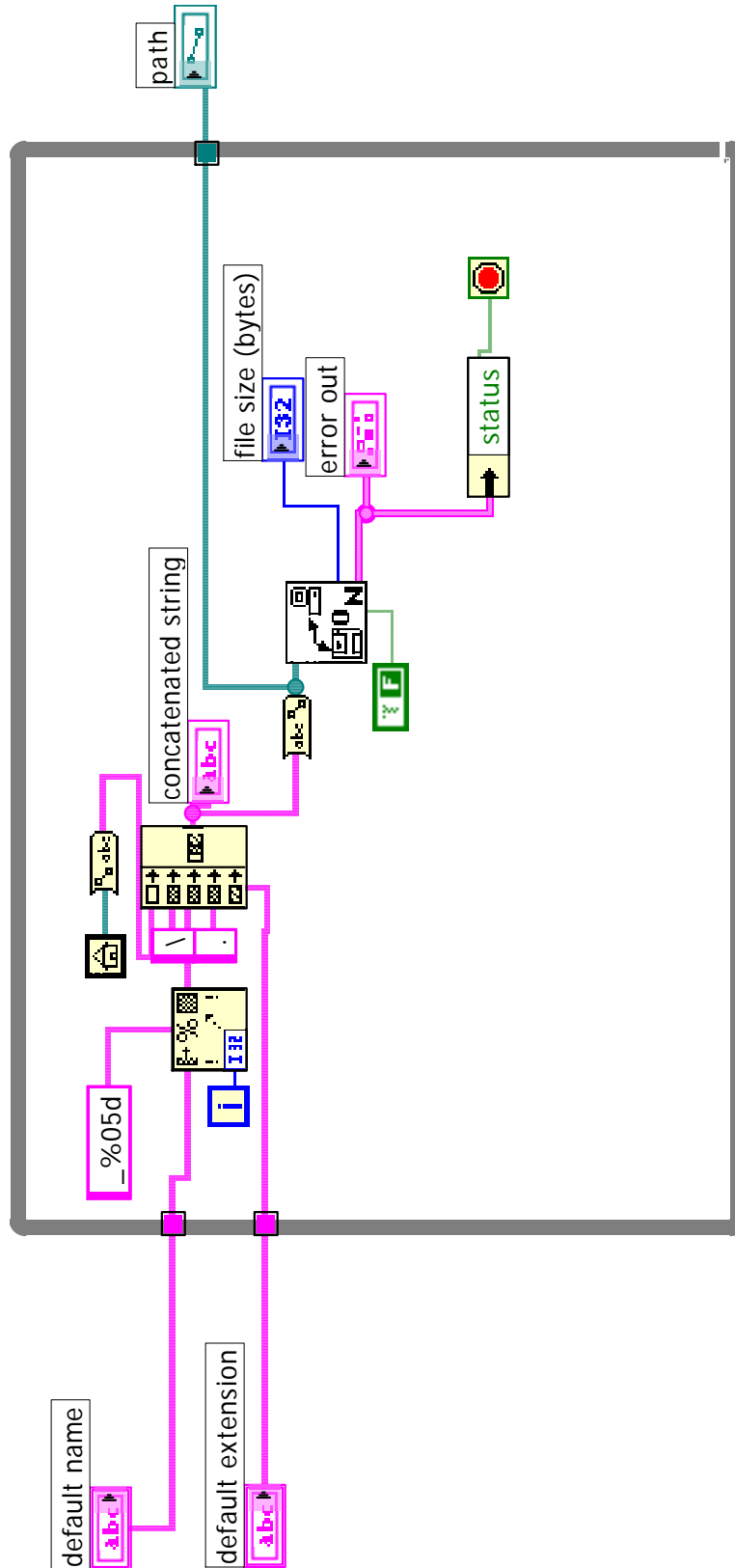


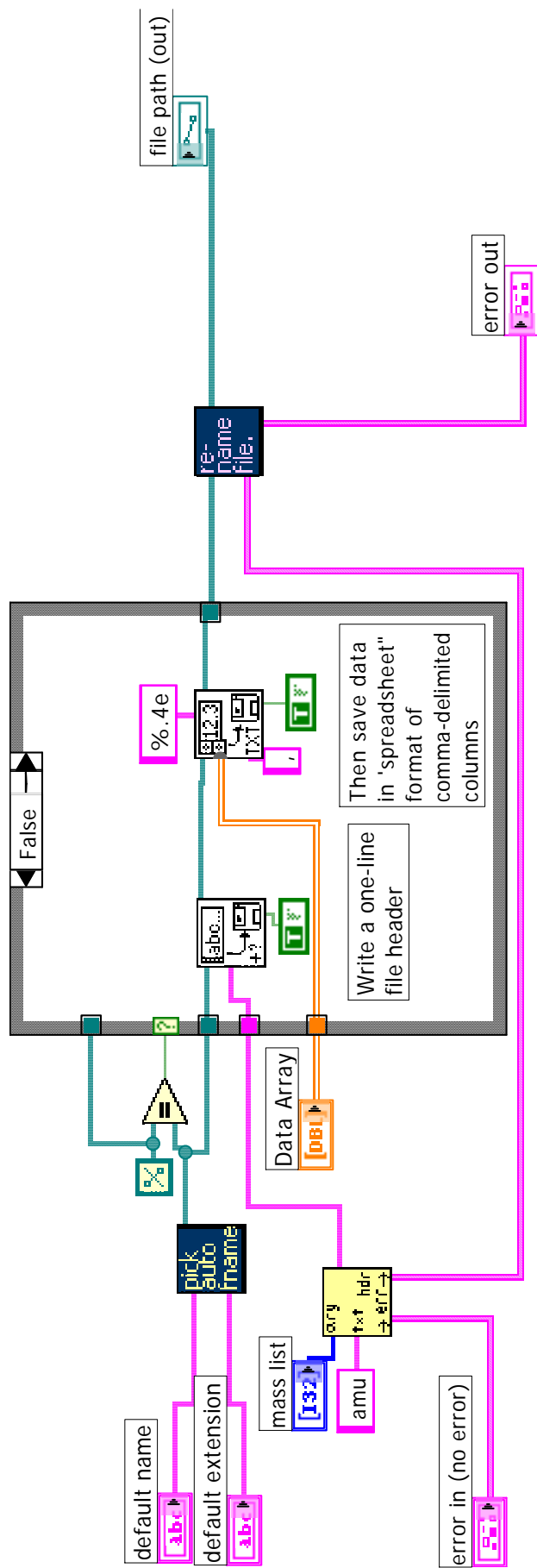








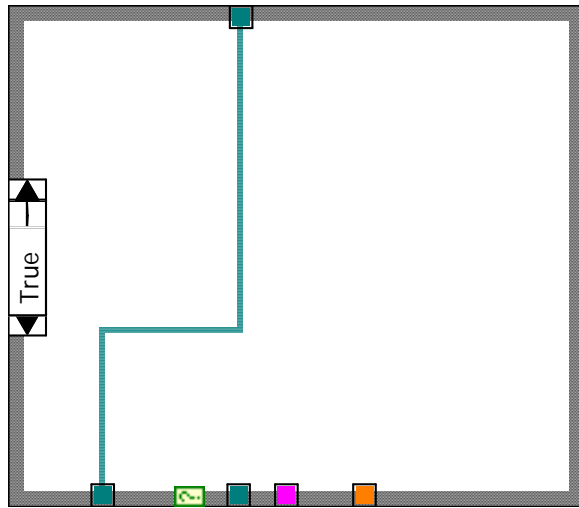




This VI first saves the data to disk in a comma-delimited ASCII format. The file is written after a file name is generated automatically using the default naming convention of AUTOSAVE_#####.TXT where ##### starts at 0 and is incremented for each successive new file.

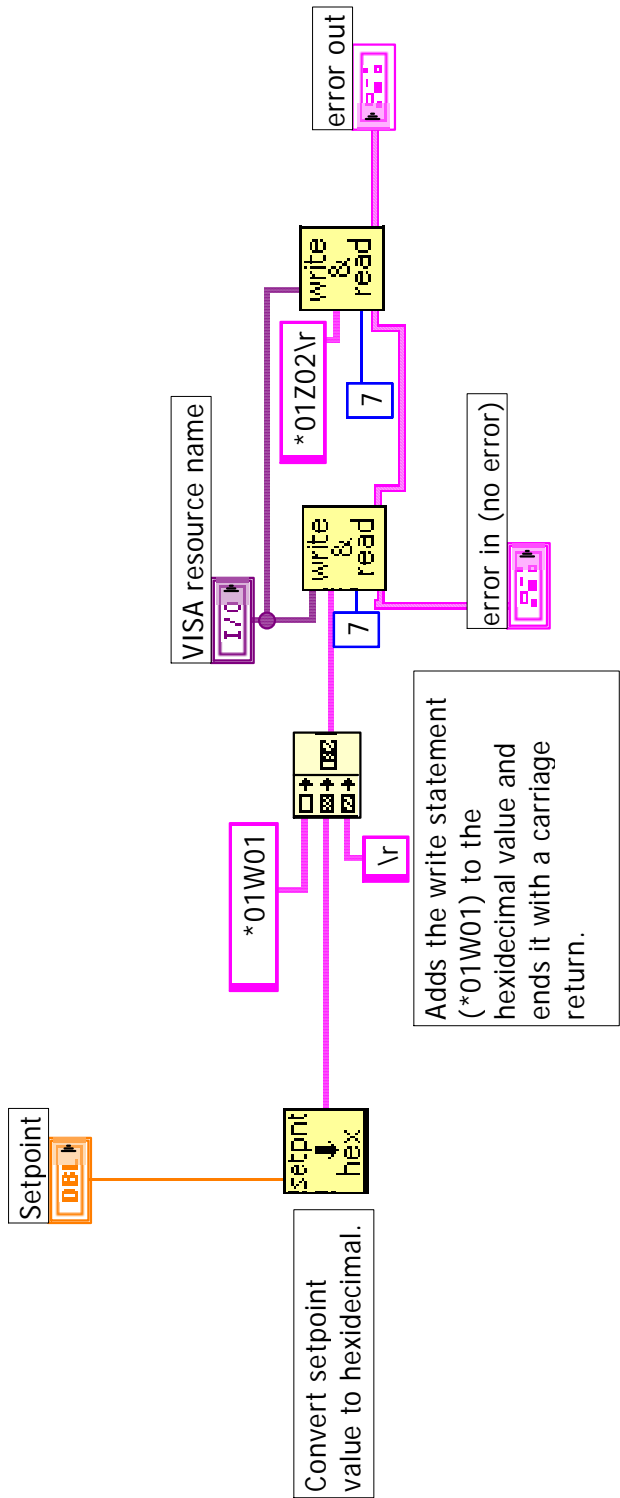
The user is prompted to rename the file, if desired. Note that the renaming consists of copying AUTOSAVE_#####.TXT to the new name (or path) and then deleting the starting file.

If no action is taken, the auto-saved file remains on disk until it is manually deleted or moved by the user using Windows tools.

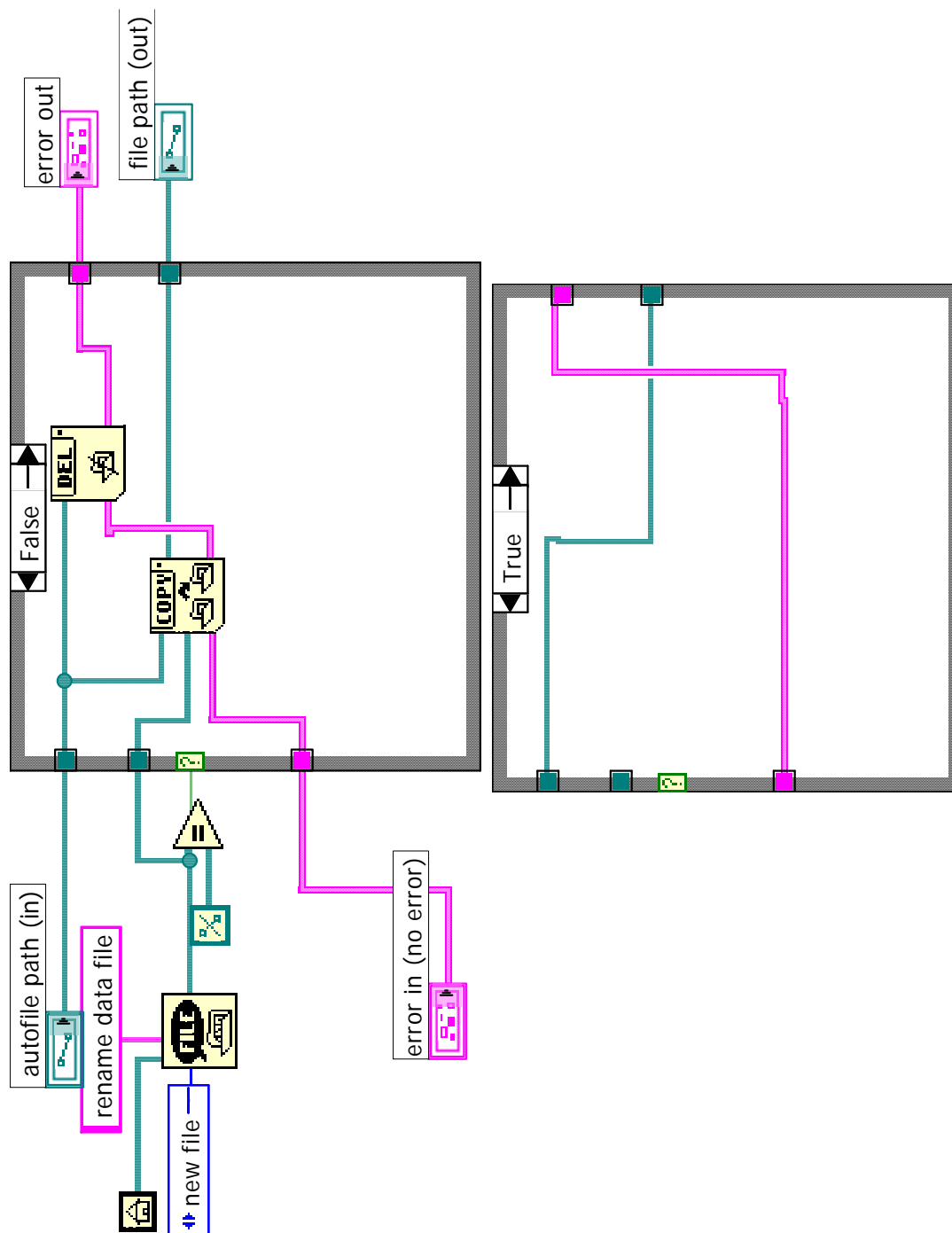


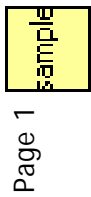
writesetpoint.vi
C:\WPDocs\Contracts\C01-07 (NASA ITD mass spec)\ITDlaptop\Ferran_MPA_IIb\ITD\writesetpoint.vi
Last modified on 12/21/2004 at 9:45 AM
Printed on 12/21/2004 at 9:45 AM

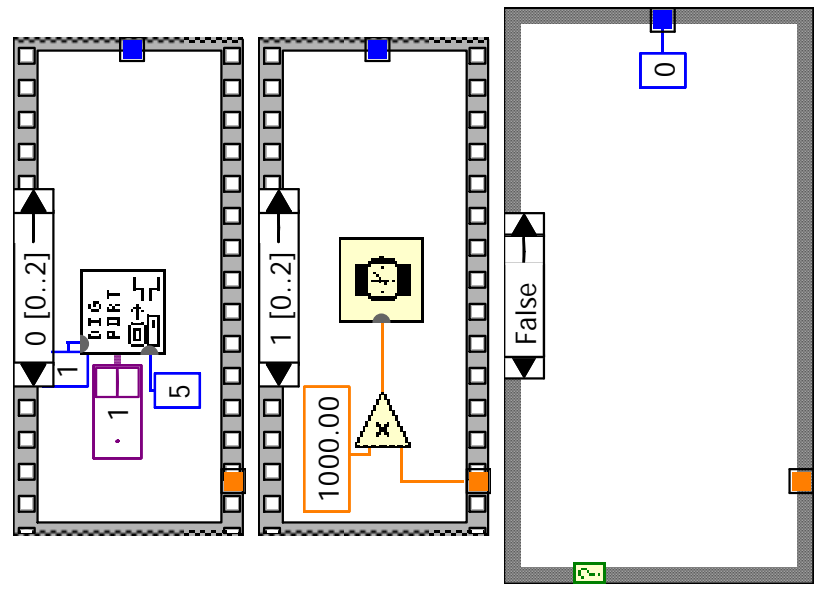
Page 1
write
setpoint

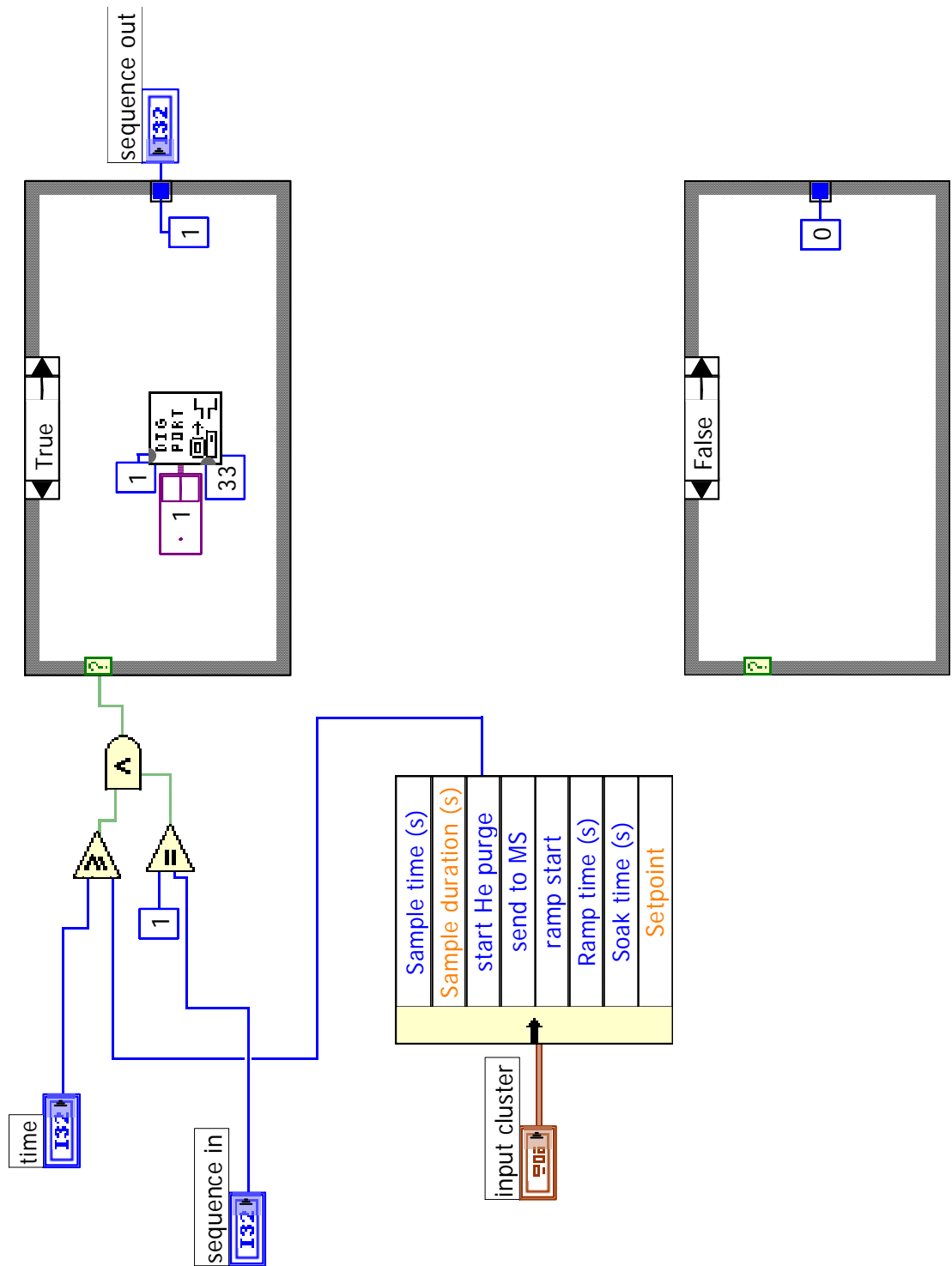


This VI writes a setpoint temperature to the Omega temperature controller in COM5 (default).







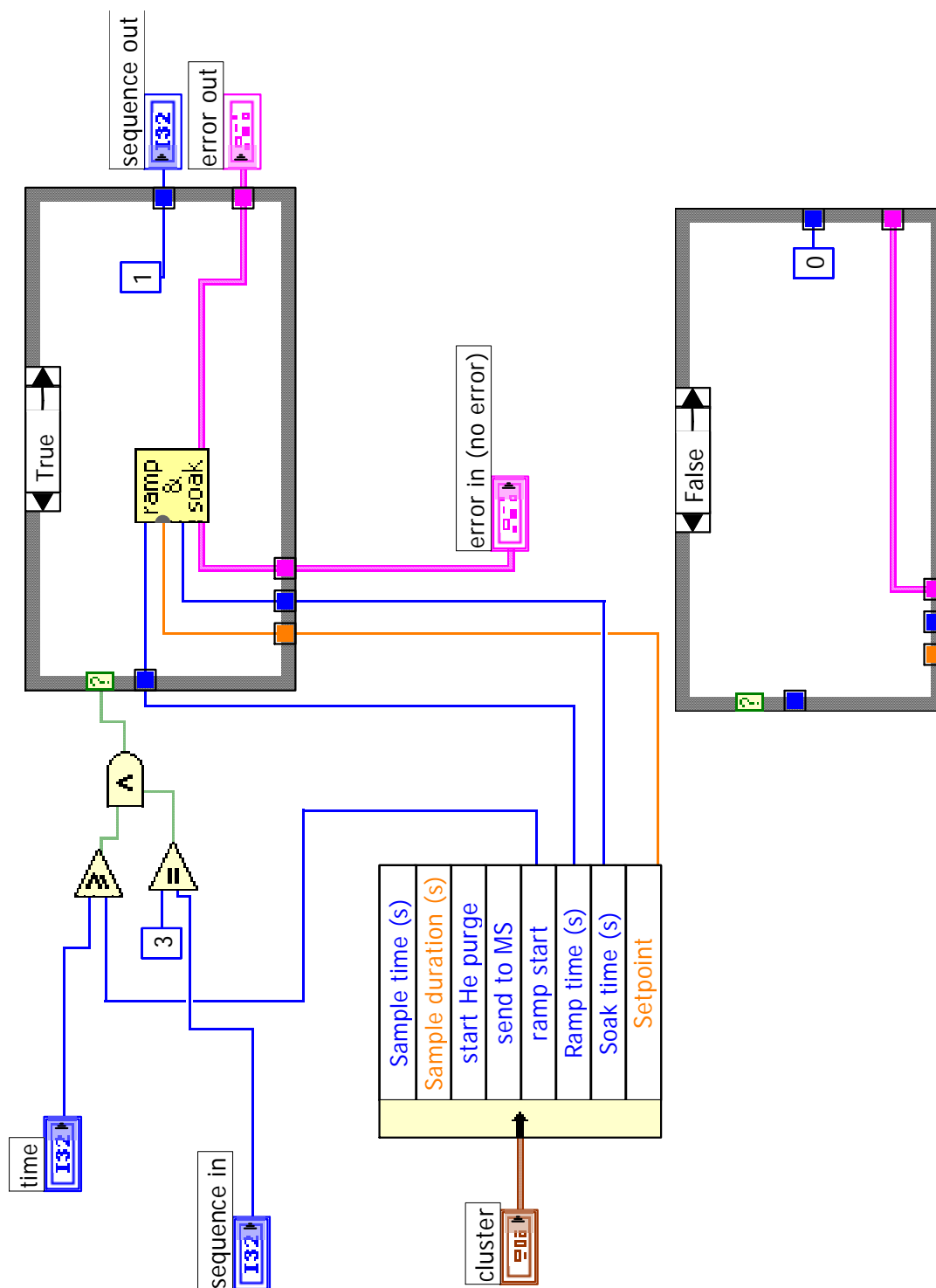


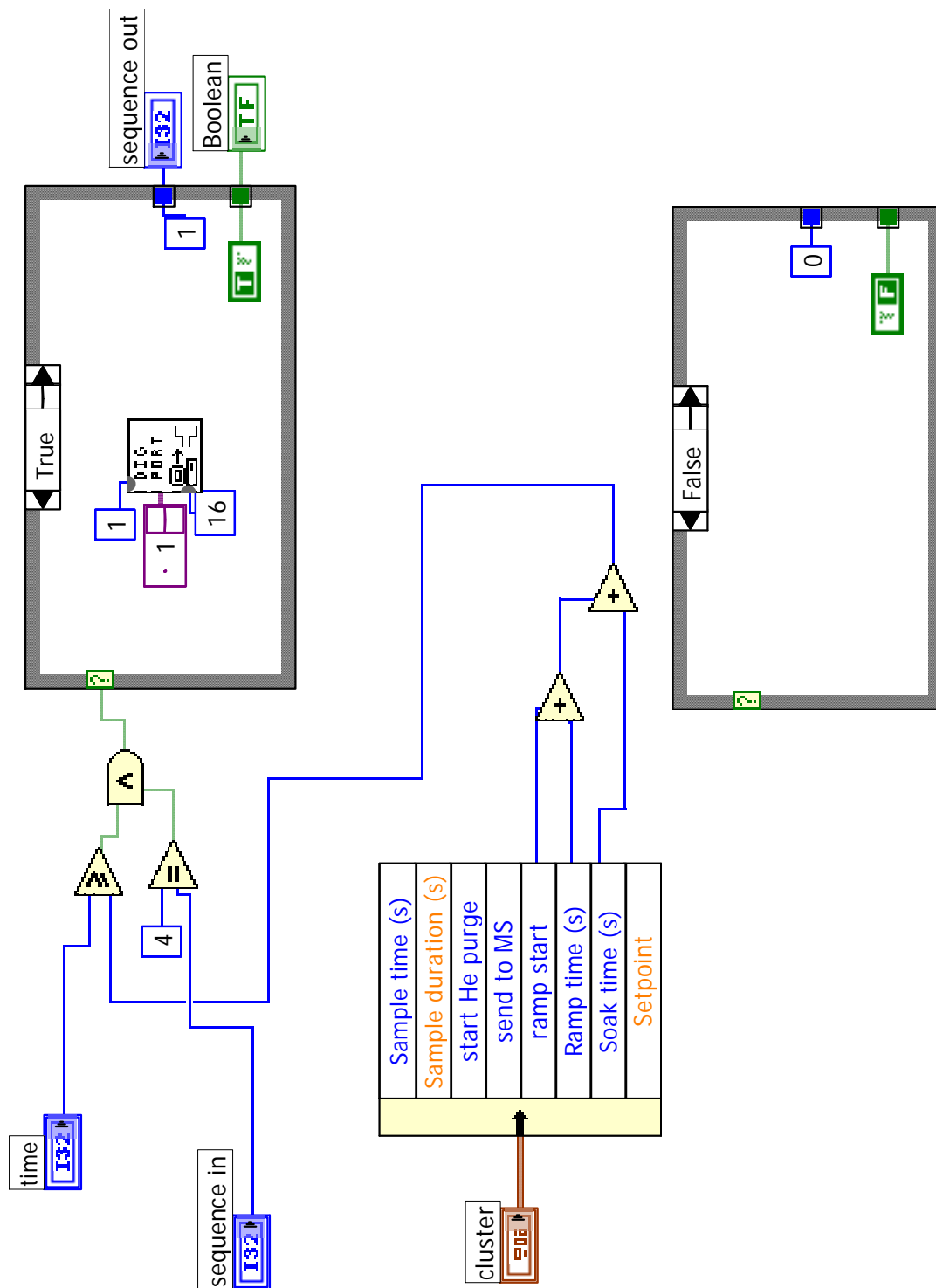


starttemp.ramp.vi

Last modified on 8/31/2004 at 2:08 PM

Printed on 12/21/2004 at 9:49 AM





Appendix B

Ferran Scientific communications protocols

CHAPTER THREE

Programming Reference

Introduction

Programming the System is accomplished by writing to registers in the CNI (or CNI) module. These registers are organized into banks of registers. The current values of various registers can be read by performing Read requests. Likewise, the current values of various registers can be changed by performing Write requests.

Here is a summary of the five banks and their general functions.

<i>SYSTEM BANKS</i>		
Bank Letter	Bank Name	Description
A	AMU Pressure	The AMU pressure bank stores the current partial pressures measured for each AMU.
P	AMU Priority	Stores numbers which express the relative priority of each AMU. Setting values in this bank allows increasing or decreasing the System's response time to various AMU's.
I	Integer Status	Stores all System status and control registers that are best expressed as nonfractional numbers.
N	Analog Pressures	Stores each data point collected during the last AMU scan. It is the information from this bank that gets accumulated and processed into the results seen in Bank A. WARNING: Obtaining information from this bank requires a magnitude more RS-232 (or RS-485) communications between the Master and the System. Reading information from this bank is not recommended unless it is completely necessary.
R	Real Status	Stores all System status and control registers that are best expressed as numbers in scientific notation.
T	Text Status	Stores all alphanumeric information. For example, the System's model number and serial number can be read from this bank. In addition, this back stores the current error message.

The details of each of these banks are described later. The next section describes the protocol used to read and write information into these banks.

Format of the Packets

This section covers the general format for all communication between the System and a Master. There is only one difference in protocol between CNI Module's operating in RS-485 mode versus RS-232 mode: The network mode requires transmitting characters in 9 bits instead of the normal RS-232 mode of 8 bits. This difference is also discussed in the following section.

Send Packets

There are two types of packets to send to the System: a Read Request and a Write Request packet.

Read Request:

RL####[:AAAA][;Comment] { CR }

Write Request:

WL####,DATA[:AAAA][;Comment] { CR }

where:

L	is replaced by a character which stands for the desired bank:
A	AMU Pressure bank
P	AMU Priority Control bank
I	Integer Status/Control bank
R	Real Status/Control bank
T	Text Status bank

The number of the register to read or write within the bank selected. Remember that all numbers must be right justified.

:AAAA An optional parameter which can be used as a packet sequence number. Each letter A stands for an ASCII letter or number. If the Master uses this option in a command, the System will send back a colon (:) followed by the exact four characters that it received. In effect, this can act as a packet sequence number that the Master can use to manage packets received.

;Comment An optional parameter which both sides can use to contain comments. In general, the comment should not be used by either side because it lowers communications speed. If register I0005 is set to 1, then the System will include comments for all errors.

,DATA A parameter that contains the information to write into the bank. Note that each bank stores a specific type of information: Integer (4 digit integer), Real (#####E+##), or Text (ASCII text).

Receive Packets

There are two types of packets that a Master can receive: an Error packet and an Answer packet.

Error packet:

```
EL####,eeee[:AAAA][;Comment]{CR}
```

Answer packet:

```
AL####,Response[:AAAA]{CR}
```

where:

eeee

The error number.

L####

The bank and register where the operation occurred.

;Comment

A brief error message. By default, the System doesn't send error messages. But, setting the register I0005 to 1 will enable error messages to be placed in this area. Please see the information on I0005 in the *Details of Banks* section for more information.

,Response

A reading of the register read or written. Normal responses are ##### for integer variables, and #.###E+## for real numbers.

NOTE: After the System performs a write, it will send a response in the same format as a response to a read. This allows the external program to confirm that the correct write took place.

Protocol of the CNI Module when in RS-485 mode

This section discusses the difference between the normal RS-232 mode and the RS-485 mode of the Computer Network Interface Module.

Four-Wire RS-485 Cable

The System CNI Modules use a four wire RS-485 cable. Two of the wires are transmutation lines from the Master. The other two wires are transmission lines from all of the CNI's that are on the cable.

Token-Type Protocol

To allow several CNI's to transmit on the same communications lines, a "token"-type protocol was established. In this protocol, all CNI's on the lines listen for a special address character that identifies to which CNI the Master is communicating. When a CNI receives its address, it automatically turns on its RS-485 transmitter and begins to listen for any commands that are

placed on the network. At the same time, all other CNI's disable their transmitters and continue to wait for the special address character.

9-Bit Serial Protocol

To help speed this process and to minimize interruptions to CNI's that are not part of the current "conversation", a 9 bit serial protocol was adopted. In this protocol, the UART of the Master (and the UARTS of all of the CNI's) enable a special 9 bit serial mode. (As opposed to a normal 8 bit serial mode.) In this mode, the normal 8 bits are transmitted plus an extra bit that the programmer can switch. This extra bit is used to mark whether a character is a special address character or a normal one.

Special vs. Normal Characters

When a 9 bit character's ninth bit is set to a 1, all CNI's use the other 8 bits as an address. When the 9th bit is set to a 0, the remaining 8 bits are read normally. For example, here is a sample message that the Master may send to the network lines:

2 WI0001,0001{CR}	'Tell CNI at 2 to turn on scan mode
AI0001,0001{CR}	'CNI at address 2 responds

The number 2 (that is in bold) is the *only* character that is sent with its ninth bit set. This command tells the CNI at address two (2) to turn on scan mode. The CNI that has its Configuration Switches set to address 2 will turn on its transmitter when it receives the 2. It will also receive the WI0001,0001 command and will respond. This CNI continues to keep its transmitter on and continues to receive and respond to commands sent by the Master to the network. The Master does not have to send the address character until it wants to address a different CNI.

In contrast, all other CNI's that receive the address 2 will turn off their transmitter (if they had it on), and will not receive or respond to the WI0001,0001 command. These CNI's will remain "dormant" until another special address character (a 9 bit character with the extra bit set) with their address is received.

NOTE: The word dormant refers to their transmitters being off. The CNI's will still continue to run normally in the mode that they were placed in. For instance, the Master could send the following sequence of packets:

2 WI0001,0001{CR}	'turn on scan mode for CNI at address 2
AI0001,0001{CR}	'CNI 2 responds to the command
3 WI0001,0000{CR}	' "turn on" idle mode for CNI at address 3
AI0001,0000{CR}	'CNI 3 responds to the command
4 WI0001,0001{CR}	'turn on scan mode for CNI at address 4
AI0001,0001{CR}	'CNI 4 responds to the command

The CNI that is at address 2 will turn on Scan Mode and will continue to run in Scan Mode even after the address 3 and address 4 are sent. Also note that the response packets from the CNI's are completely normal and do not include a network address. (They will be transmitted in 9 bits however.)



WARNING: If you set the Configuration switches so that two or more CNI's have the same address, they will all turn on their transmitters and garble each others' responses.

One final note, the Master should not send a new address to the network until a CNI has finished responding to its last given command. Doing so can cause the response to be garbled.

Details of the Banks

General Notes

All of the information that an external program needs to read or write is contained in a register of one of the banks. These sections detail what each of the registers control or report.

Before continuing, there are a few notes to point out about register numbers and reading from or writing to register numbers that don't exist.

- All register numbers are four digits long. Register numbers for some of the banks have their first digit coded. (The Integer, Real and Text banks are examples.) A 1 as the first digit means the register is READ ONLY. A zero or any other number means that it can be written to or read from. For example, bank T, the Text Status Bank, has a register 1001 that stores the manufacturer's name. This register, because its first digit is 1, is read only.
- When you read from or write to a register that doesn't exist, you will receive a communications error message. Depending on how you write your communications software, this can lead to a lock-up. As a short explanation, you may write software that unintentionally asks for an AMU that doesn't exist. The Micropole firmware will respond with a communications error. Your software may be designed to retransmit the packet if any communications errors occur. This, of course, causes the firmware to send the same communications error. This process could then repeat forever. You can write some error checking code to make sure that your software does not do this. See registers I1005 and I1006 for information on the System's capabilities.

AMU Pressure Bank (BANK A)

The AMU pressure bank stores the current partial pressure (in Torr) of each of the AMU's the System has measured. The registers are read only. Register number one (1) corresponds to AMU number one (1). The System does not report partial pressures of fractional AMU's. (See the Analog Pressure Bank for more information.)

General Format:

RA####{CR}

Example:

RA0040{CR} 'Read the partial pressure of AMU 40.

AA0040,1.231E-03{CR} 'a possible response to the command to read Ar (AMU 40)

Read requests of an AMU number that is beyond the abilities of the analyzer will get a zero response or an error message. To help the external software, the minimum and maximum AMU's can be obtained by reading registers I1005 and I1006.

All registers in this bank default to 0.000E+00 Torr upon power up, reset or when the System is first changed to a non-idle mode.

AMU Priority Bank (BANK P)

This bank roughly controls the amount of time spent on each AMU that the MPA can measure. Each of the registers represents one AMU and contains an integer from 0 to 2. The System scans registers (AMU) with 2's twice as often as AMU's marked with 1's. AMU's marked with 0 receive no scanning whatsoever.

You can increase the scan speed of the System significantly by setting the priority of the gases that you are not interested in with a 0.

Here are some examples:

WP0040,0002{CR} 'Make AMU 40 a higher priority
AP0040,0002{CR} 'AMU 40 now has priority 2.

WP0006,0000{CR} 'Do not scan AMU 6
AP0006,0000{CR} 'AMU 6 has priority 0 and won't be scanned

This bank defaults to values of 1 when the System is powered up or reset. You can change the values of all of the registers in this bank with a single write to register I0020. Please see the appropriate section for more information.

Integer Status Bank (BANK I)

This bank controls the various options of the System. It also contains information that is most conveniently stored as an integer. The power up and reset values of the register are indicated with the word default in parentheses.

CHART OF BANK I REGISTERS

Register Number	Name	Description/Value
I0001	Device Mode	0 = Idle (default) 1=Scan Mode (continuous) 2=Leak Detection Mode 3=Calibration Mode (not yet available) 4=Scan and Hold Mode 6=Jump Scan 9999=Internal Test Mode. Used for factory testing of the CNI. DO NOT PUT THE SYSTEM INTO THIS MODE: System can be

CHART OF BANK I REGISTERS

Register Number	Name	Description/Value
		damaged.
I0002	Reset Device	0=No (default) 1=Perform full reset. All registers are returned to their power-up defaults
I0003	Degas Mode	0=Degas Off (default) 1=Degas On. e Energy Mode and the filament emission is turned to High.
I0004	Electron Energy Mode	0=Low, 42V (default) 1=High, 68V (Not Available)
I0005	Verbose Error Messages	0=No error message text sent. (default) 1=All error messages will be followed by text describing the error. NOTE: You can get the error message text by reading the T1005 or T1007 register.
I0006	Auto Range Mode	not used. Reserved for future
I0007	Leak Detection Gas	not used. Reserved for future.
I0008	Calibration Gas	not used. Reserved for future.
I0009	Automatic Shutoff Period	0=Automatic shutoff deactivated. (default) Any other number written here activates the automatic shutoff. If you read this register, it will return the number of minutes left before shutoff.
I0010	Continue Scan	1=Put In Hold 0=Release From Hold (default) Used only with Scan Once and Hold. After every scan, the System sets this register to a one (1). The System doesn't begin another scan until a zero (0) is written here by the Master.
I0012	Electrometer Speed Select	not used. Reserved for future
I0013	Range switch Speed select	not used. Reserved for future.
I0014	AMU Cal Speed select	1 = Fast; 1 = Medium; 5 = Slow; 10 = VSlow
I0015	Analyzer EmissM	This is a MPA calibration factor. It should not be changed from the value that the factory has given.
I0016	Autozero mode	0=enabled 1=disabled not used. Reserved for future.
I0017	Start scanning at AMU	0=Start scanning at the minimum AMU the System can measure (default) A non-user number is treated as the first AMU to measure in a scan.
I0018	End scanning at AMU	0=End scanning at the maximum AMU the System can measure (default) A nonzero number is treated as the last AMU to measure in a scan.
I0019	Total Pressure Mode	0=off (default) 1=on
I0020	Set Priority Bank	Sets the priority bank to the value written here.
I0021	Version	Acceptable power level 6,7,8, and 9 (no check)
I0022	CNL I/O	Logic Function Enabler
I0023	Force	0=Automatic(default); 1=Filament A; 2=Filament B

CHART OF BANK I REGISTERS

Register Number	Name	Description/Value
	Filament	
I0024	# of Samples for OSC mode	
I0025	Mux Position for OSC mode	
I0026	User Specified # of points / AMU	
I0027	Hold Filament ON	0=off (default) 1=on
I0028	Hold More Point Scanning	0=Fast; 1=Medium, Slow, VSlow
I0029	Dwell Time	Reserved for future.
I0030	E. Multiplier	Reserved for future.
I0031	Load NOVRAM	Reserved for future.
I0032	Low Gain Mode	Lock Electrometer in VFast Scan. Reserved for future.

READ ONLY REGISTERS

I1001	Error Code	0=No errors occurred. (default) Any other number represents an error. See the section on Error Codes for more information. The text of this error is stored in register T1005.
I1002	Scan Time	not used. Reserved for future.
I1003	Scan Counter	This register counts the number of scans that have occurred. This gets updated at the end of each scan and can be used to tell when a scan has been completed
I1004	Last AMU Processed	not used. Reserved for future
I1005	Lowest AMU	Lowest AMU that the System can measure using the SC and MPA connected. WARNING: This register is valid only when an SC is connected to the CNI or CNI. (Defaults to 0 if no SC connected.)
I1006	Highest AMU	Highest AMU that the analyzer can measure using the SC and MPA connected. WARNING: This register is valid only when an SC is connected to the CNI or CNI. (Defaults to 0 if no SC connected.)
I1007	Maximum Number of Filaments	Total number of filaments on the MPA.
I1008	Number of Usable Filaments	Number of filaments on MPA that are available for use.
I1009	Filament	0=All filaments are off 1=Filament A is in use 2=Filament B is in use

	Status	
I1010	Number of Electrometer Ranges	Not used. Reserved for future.
I1011	Communications Error Code	0=No Errors. (default) Any other number represents an error code. See the section on Error codes for more information. The text of this error is stored in register T1007.
I1012	Pressure Threshold Trip Status	No used. Reserved for future. See R0004 for more information.
I1013	Current AMU Reading	This register stores the AMU numbers that are currently being scanned by the System.
I1014	SC Code	Resistor Code

Real Status Bank (BANK R)

This bank contains the various configuration options of the MPA. It also contains information that is most conveniently stored as a real number.

<i>CHART OF BANK R REGISTERS</i>		
Register Name	Name	Description/Value
R0001	Manometer Span, Volts/Torr	0=No manometer connected (default) A zero (0) written here means that the manometer will not be read or used for filament protection. Otherwise, the span of the manometer as expressed in Volts/Torr is stored here.
R0002	Manometer Zero Offset	0=No manometer zero offset (default) This stores the zero offset of the manometer, expressed in Torr. the System will then subtract this number from all readings of the manometer.
R0003	Pressure of Calibration Gas	Not Used. Reserved for future.
R0004	Threshold Trip Pressure	Not Used. Reserved for future.
R0005	Analyzer Sensitivity Factor	611 (default) The Sensitivity calibration factor provided by the factory for the Micropole Analyzer currently connected to the System.
R0006	Analyzer Ion Adjust	12 (default) The Analyzer ion calibration factor provided by the factory for the MPA currently connected to the System.
R0007	Analyzer AC Adjust	6.6 (default) The Analyzer AC calibration factor provided by the factory for the MPA currently connected to the System.
R0008	User Sensitivity Adjust	1.000 (default) A multiplier for sensitivity of the analyzer. If the System reports a Nitrogen peak of 1 mTorr when it should read 2 mTorr, a two (2) can be written here to adjust the reported sensitivity.

CHART OF BANK R REGISTERS

Register Name	Name	Description/Value
R0009	Analyzer AMULOC	1.000 (default) The calibration factor provided by the factory for the Micropole currently connected to the System.
R0010	Analyzer TSense Adjust	6000 (default) The calibration factor provided by the factory for the Micropole currently connected to the System.
R0011	Electron Energy	Reserved for future.
R0012	Calibration Frequency	Reserved for future.

READ ONLY REGISTERS

R1001	Total N2 Equiv Pressure Reading	The current reading of total pressure. Only updated when total pressure mode is on. See 10019 for more information.
R1002	Manometer Reading	The current reading of the manometer, in Torr
R1003	Lowest Detectable Pressure	The lowest pressure the System can detect, in Torr.
R1004	Highest Detectable Pressure	The highest pressure the System can detect, in Torr.
R1005	Highest Pressure Before Shutdown	If the pressure exceeds this value, the System will turn off the filaments and return to the idle mode.
R1006	SC Temperature (C)	Obsolete. Was used for CNI's to report current temperature of the SC Module.
R1007	Max SC Temperature Allowed	Maximum rated temperature for the SC Module.
R1008	Fil. A ON Time	0.1h Granularity
R1009	Fil. B ON Time	0.1h Granularity

Text Status Bank (BANK T)

This bank stores information and messages about the System. It also contains information that is most conveniently stored as a text.

CHART OF BANK T REGISTERS		
Register Number	Name	Description/Value
READ ONLY REGISTERS		
T1001	Manufacturer's Name	Contains the name of the factory that created the CNI Module.
T1002	CNI's (or CNI's) Model Number	Contains the CNI Modules' model number.
T1003	Serial Number of CNI (or CNI)	Not Used. Reserved for future.
T1004	Firmware Version	Contains the Firmware version number.
T1005	Last Error Message	This will have the last error message. (Note: Communications errors are not recorded here.)
T1006	SC's Model Number	Contains the model number of the SC that is currently connected to the CNI Module.
T1007	Last Communications Error	This will have the last communications error message.
T1008	SC S/N	Reserved for future.
T1009	MPA Model	Reserved for future.
T1010	MPA S/N	Reserved for future.

Analog Pressure Bank (BANK N)

This bank stores the actual point-by-point readings of the System. The registers are read only and their contents are an (X,Y) pair of coordinates for the data point read. Unlike Bank A, the register number refers to the number of the data point and not the number of the atomic mass unit.

An (X,Y) pair that has X=0 marks the end of the data points for the scan run. Otherwise, the X value is the AMU number in the format of ###.#. The Y value is the partial pressure (in Torr) measured at the AMU X. Here is an example:

```
RN0001{CR}           'give me the data point number 1
AN0001,002.4,1.120E-03 'partial pressure read at AMU 2.4
```

To use this bank, you need to keep reading data points until you encounter one that has an X value of 0. For this reason, the length of the packet, and the number of data points in a typical scan, it is NOT recommended that you read this information. See the *Optimization of Results* section of this manual for more information.

Appendix C

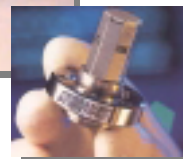
Ferran Scientific data sheet



FERRAN SCIENTIFIC

Affordable Gas Measurement, Control and Analysis

Symphony Micropole™ Mass Spectrometer System



CE

- ◆ Faster Scan Rates (600 ms / amu)
 - ◆ 10^{-10} Torr Limit of Detection
 - ◆ **10 parts per billion Sensitivity**
 - ◆ 43 eV or 70 eV Ionization Energy Settings
 - ◆ 2×10^{-8} atm.cm³/s Minimum Detectable He Leak Rate
 - ◆ New PlasmaGuard Extends Sensor Life

New

FSInc. March - 2002

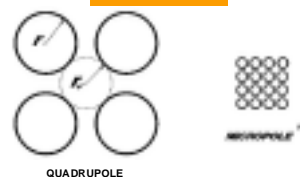
Symphony is the world's smallest complete mass spectrometer system. The small size, low cost, and component interchangeability provide the user with a set of powerful features and unique advantages. The mix-and-match feature is crucial for easy upgrade, troubleshooting, and replacement. In addition, the ability to operate at higher pressures (mTorr range) without sacrificing UHV measurement capabilities translates into a two-fold advantage: measuring gaseous concentrations directly using an open source and reducing or eliminating additional vacuum pumps. Since the vacuum pumping requirements are less stringent, **Symphony** systems can be packaged together with various sample introduction methods in the form of portable instruments for field applications.

- ◆ *Small Size*
- ◆ *Low Cost*
- ◆ *Interchangeable Components*
- ◆ *Pre-calibrated, Pre-tuned Sensors*
- ◆ *High Pressure Operation*
- ◆ *Easy-to-use*



At the heart of the **Symphony** is an enhanced version of the Micropole™ (U.S. Patent. 5,401,962) mass analyzer, the **PlasmaGuard**. Its breakthrough design using a miniature array of quadrupole mass filters revolutionized the world of mass spectrometry. The Micropole offers a similar or greater sensitivity than conventional mass spectrometers, but in a fraction of the volume. Its fabrication (U.S. Patent. 5,857,890) uses cutting edge technologies such as highly accurate photo etched components and proprietary glass-to-metal seals. The mass production of these sensors translates into a lower manufacturing cost. Connected to the PlasmaGuard is a state-of-the-art miniature, low power, high frequency power supply. Since these units are factory pre-tuned, they are interchangeable. The sensors are pre-calibrated against transfer pressure standards in order to provide direct absolute **total** and **partial** pressure measurements (in Torr). Through a computer interface, the instrument is controlled using MS Windows compatible software (WinMPA). Automatic, around the clock data acquisition in different formats is built-in for fast access to the information required from the mass spectrometer.

Array Concept



Operation of a Single Quadrupole of the Micropole



Symphony Components:

Micropole and PlasmaGuard Analyzers:

The Micropole (MPA) is a miniature array of quadrupole mass spectrometers designed to operate simultaneously (in parallel). Atoms and molecules are ionized in a single Nier-type ion source following electron impact (EI). Electrons are emitted from hot filaments located outside the field of view (main axis) of the quadrupole detector. Using electrostatic lenses, ions are extracted, focused, and injected into individual quadrupole mass filters. Ions with a specific mass-to-charge ratio are filtered by applying a combination of DC and RF voltages to each quadrupole in the array. Filtered ions in each quadrupole are collected on individual Faraday type detectors. All individual detectors contribute to generate a single current signal. A mass spectrum is generated by scanning both AC and DC voltages. A MPA chassis includes a total of 16 rods and 11 electrical pins all secured in a glass-to-metal structure. The structure is sealed in a high temperature oven. The electrostatic lenses, apertures, and plates are made of highly accurate photo etched parts. The sensor construction does not use ceramic parts as is the case with conventional quadrupoles. Therefore, no charging effects are observed.

The **Symphony** system features a more robust version of the Micropole: the **PlasmaGuard**. It is encapsulated in a stainless steel case for easy handling. The analyzer is also protected by a mesh, on the vacuum fitting, designed to extend the lifetime of the sensor in harsh environments such as plasma processes. Each MPA/PlasmaGuard sensor is delivered with a set of six calibration parameters generated at the factory for fast and easy operation. Two ionization energy settings are built into the **Symphony** system: 70 eV and 43 eV. While 70 eV is standard, 43 eV setting is used to avoid spectral interferences from doubly charged ions. For example, the water peak at ($m/e = 18$ amu) is free from $^{36}\text{Ar}^{++}$ interferences since, 43 eV is just below the second ionization potential of ^{36}Ar .



Spectra Converter (SC):

The SC includes a pre-tuned RF power supply and an extremely sensitive electrometer circuitry. Using a built-in guide pin, the SC is easily connected and secured to the back of the MPA/PlasmaGuard with a connector latch. The combination can be mounted in any orientation. MPAs/PlasmaGuards and SCs are interchangeable for fast and easy replacement, upgrade, and troubleshooting.

Computer Network Interface (CNI):

The SC is connected to the CNI using a shielded cable. The cable is immune against surrounding electronic noise from other equipment. The CNI includes the power supplies, the control circuitry and the communications board. All circuits are driven by an internal microcomputer and embedded control software (firmware). The CNI is directly connected to a PC via RS232, RS485, or TCP/IP serial interfaces. For multiple sensor installations, an RS232/RS485 converter is on board. The CNI is powered by a 24 VDC power supply.



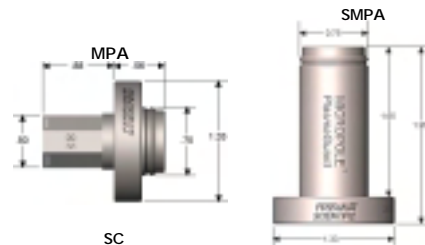
Computer Network Logic Interface (CNL):

In addition to the CNI circuitry, the CNL includes a board with digital Input/Outputs and analog output capabilities. A standard Capacitance Diaphragm Gauge (CDG) connection is also available. The additional circuitry enables the user to implement fault detection scenarios using custom decisions based on the data acquired. Automatic operation using external triggers is easily set. Connecting a CDG or other equivalent gauge to the CNL to work in conjunction with the RGA allows the user to implement warning, automatic operation, and sensor protection features.

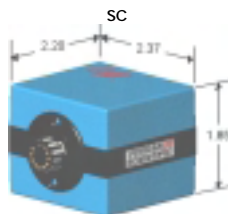


Symphony Specifications:**Micropole and PlasmaGuard Analyzers (MPA and SMPA):**

- Dimensions: see drawing
- Weight: 100 g
- Twin-Filament Electron Impact Ion Source
- Filaments: Y_2O_3/Ir (standard), $ThO_2/Ir, W$
- 9 Miniature Quadrupole Mass Filters
- Mass Filters: 2, 5, 10, 15, 20 mm Long
- Faraday Cup Detector
- Max. Bake out Temperature: 350 °C
- Max. Operating Temperature: 150 °C
- UHV Compatible: $< 10^{-9}$ atm.cm³/sec Rate
- Mounting Flange: mini-Conflat (standard), NW/KF16

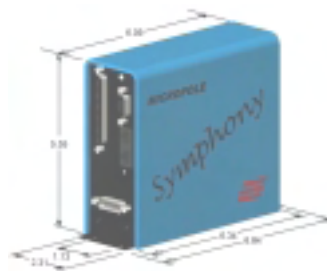
**Spectra Converter (SC):**

- Dimensions: see drawing
- Weight: 200 g
- RF Power Supplies: 7, 11, 14, or 18 MHz
- Factory Pre-Tuned
- Interchangeable with any MPA/SMPA Analyzer of the Same Type
- Connector Latch for Mounting in Any Orientation

**Computer Network Interface (CNI):**

- Dimensions: see drawing
- Weight: 750 g
- Power Requirements: 24 VDC, 1.5 A
- RS232 (50 ft Max.), RS485 (3000 ft Max.) Interfaces
- RS232/RS485 Converter
- Selectable Baud Rates: 9600, 19600, 38400
- Up to 8 Units can be Daisy-Chained
- Built-in standard connections to heated and non heated Capacitance Diaphragm Gauges (See **TRIO** Brochure)

CNI

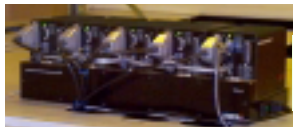
**Computer Network Logic Interface (CNL):**

- Dimensions: see drawing
- Weight: 1 kg
- Power Requirements: 24 VDC, 1.5 A
- RS232 and RS485 Interfaces
- RS232/RS485 Converter
- Selectable Baud Rates: 9600, 19600, 38400
- Up to 8 Units can be Daisy-Chained
- 8 Open Collector Logic Input/Outputs
- Standard Connector for non Heated CDGs
- 2 Pneumatic Valve Actuators
- Optional 3 Analog Outputs (0-1V)

CNL

**Accessories (See Catalog/Price List for Part Numbers):**

- 24 VDC Power Supply (35 and 65 W)
- Micropole Cable (1, 10, 15, 25 ft max.)
- RS232 Cable (6, 10, 25, 50 ft Max.)
- RS485 Cable (1- 3000 ft Max.)
- RS232/RS485 Converter
- Isolation Valves (manual and Pneumatic)
- Flange Adapters and Mounting Kits
- Grounding Strap (3 ft max.)



Micropole Docking Station for 5 Sensors (PAC-05): multi-chamber solution where power supplies, driving electronics, and wires are enclosed in a single platform.

Ferran Scientific Inc.
11558 Sorrento Valley Rd.
San Diego, CA 92121
Ph: (858) 792-2332
Fax: (858) 792-0065
Web Site: <http://www.ferran.com>
E-mail: sales@ferran.com

Symphony Options:

PlasmaGuard (SMPA)	SMPA-1-2/45-X ²	SMPA-11-2/45-X	SMPA-5-2/65-X	SMPA-7-2/65-X	SMPA-5-2/100-X	SMPA-1-4/300-X
Micropole Analyzer (MPA)	MPA6-1-2/45-X	MPA6-11-2/45-X	MPA6-5-2/65-X	MPA6-7-2/65-X	MPA6-5-2/100-X	MPA6-1-4/300-X
Spectra Converter (SC)	SC6-18	SC6-18	SC6-14	SC6-14	SC6-11	SC6-07
Mass Range (amu)	2-45	2-45	2-65	2-65	2-100	4-300
Resolution FWHM (amu)	0.5	1.5	0.8	0.9	1	1.5
Maximum Operating Pressure ¹ (Torr)	1x10 ⁻³	1.1x10 ⁻²	5x10 ⁻³	7x10 ⁻³	5x10 ⁻³	1x10 ⁻³
Minimum Detectable Partial Pressure ¹ (Torr) for Slow Scan Updates	10 ⁻¹⁰	10 ⁻¹⁰	10 ⁻¹⁰	10 ⁻¹⁰	10 ⁻¹⁰	10 ⁻¹⁰
Fastest Scan Updates / amu (sec)	0.6	0.6	0.6	0.6	0.6	0.6
Minimum Detectable He Leak Rate (atm.cm ³ /sec)	2x10 ⁻⁸	2x10 ⁻⁸	2x10 ⁻⁸	2x10 ⁻⁷	2x10 ⁻⁸	2x10 ⁻⁸

Ordering Information: A desired mass range requires specifying a combination of MPA/SMPA and SC. Please contact the Sales department for prices and delivery

(1) Maximum Operating Pressure Minimum Detectable Partial Pressure are specified for molecular Nitrogen
 (2) X specifies the vacuum flange type: C for 1.33" Conflat and K for NW/KF16

**Total Process Monitoring and Control Solution**

- ◆ SMPA-5-2/65C: PlasmaGuard (5 mTorr Upper Pressure limit, 2-65 Amu mass range, mounted on mini Conflat Flange)
- ◆ SC6-14: 14 MHz Spectra Converter
- ◆ CNL-06: Computer Logic Interface
- ◆ PS-03: 24 VDC Power Supply
- ◆ CA-06-XX: SMPA Shielded Cable
- ◆ CA-02-YY: RS232 Cable
- ◆ Control Software (WinMPA2002)
- ◆ VG-1A (1 Torr CDG; See TRIO Brochure)
- ◆ V12: Optional Pneumatic valve

Contact Sales Offices for Prices

XX: Cable Length = 6, 10, 15'
 YY: Cable Length = 6, 10'

Shipping Information:

Weight 5 kg Approx.
 Packaging Dimensions 45x40x10 cm



For turnkey solutions, please refer to the **Quartet** brochure available on Ferran.com. The Quartet system, AC line powered, includes Symphony components, data display and control monitor.

For Miniature, low cost, reliable CDGs, please refer to the **TRIO** brochure available on Ferran.com.



WinMPA™ Control Software:

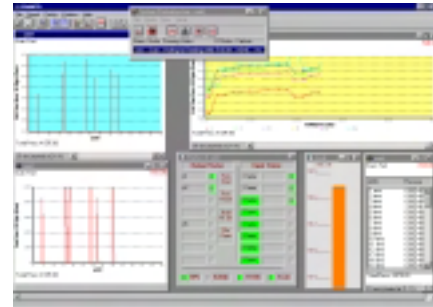
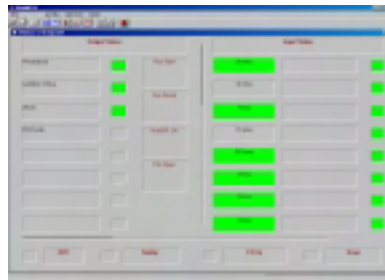
Symphony is controlled using **WinMPA**, a Windows application developed by Ferran Scientific Inc. In addition to the communication protocols, WinMPA includes data acquisition, display, and file handling capabilities. When executed for the first time, WinMPA performs a **three-step** configuration procedure. **1. ANALYZER CONFIG.:** consists of entering the calibration parameters of each MPA/SMPA. The parameters of the analyzer are stored under a name (e.g. serial number) specified under NEW ANALYZER. **2. SYSTEM CONFIG.:** a SYSTEM comprises a MPA/SMPA, a SC, and CN/CNL. Under NEW SYSTEM, the model and the serial number of the SC and CN/CNL are entered. The MPA/SMPA is selected from a pull-down menu showing all analyzers configured under ANALYZER CONFIG. **3. COMM CONFIG.** consists of linking a SYSTEM to a communication port of the PC. After setting the baud rate, the serial interface (RS232 or RS485) is selected. For multiple sensor applications, each SYSTEM has an address (0 through 32). A SYSTEM STATUS window enables the user to control, open graphs, save data, and generate process recipes for each SYSTEM independently.



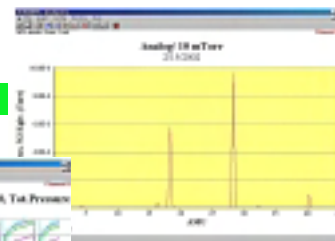
WinMPA Highlights

- Compatible with Windows XP, 2000, Me, NT 4.0, 95/98, 3.1/3.11
- PC Requirements: 66 MHz, 16 MB RAM. Higher specifications are required for multiple analyzer installations.
- Embedded demo mode
- Capture of all data all the time in temporary files
- Automatic 24/7 data capture and file naming
- Binary and ASCII data file formats
- Dynamic Data Exchange (DDE) links with spreadsheet software.
- Optional TCP/IP protocol
- Data display in multiple, simultaneous BAR, TREND, ANALOG, TABLE, and DEVIATION graphs.
- Torr, Pascal, Bar, and PPM Units.
- Linear and Logarithmic Scales
- LEDs to show status of Input/Outputs
- Monitor and display signals from complementary gauges such as CDGs.
- Custom warnings following drifts from set tolerances
- Storage of process control recipes
- Statistical process control (SPC) incorporated into recipes
- Deviation graphs showing drifts from "ideal" runs
- Event-driven full automated operation and data storage
- Jump/Continuous Scans with or without Total Pressure measurement
- Pre-set scan speed mode and graph recipes
- Sensor accumulated hours of operation
- Helium leak mode with progressive display graph and audible warning

Multi-chamber Control



Analog Scan



Trend Mode

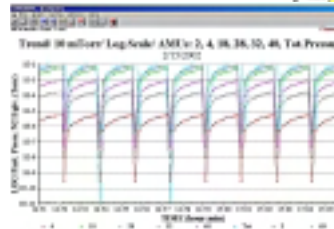
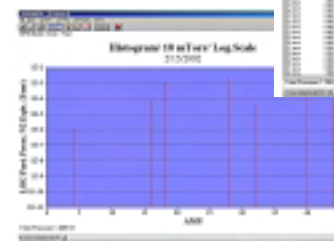


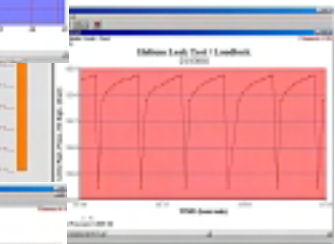
Table Mode



Histogram



Leak Mode



Deviation Graph





Symphony Typical Applications

General Residual Gas Analysis:

- ◆ Vacuum diagnosis from UHV to low vacuum
- ◆ Troubleshooting leaks
- ◆ Monitoring and control of contaminants
- ◆ Helium leak detection

Semiconductor Chip Manufacturing:

- ◆ Photoresist detection
- ◆ Moisture & contaminant detection in wafer Handling chambers of cluster tools
- ◆ Wet clean recovery after PM cycles
- ◆ Leakback test and leak troubleshooting
- ◆ Process monitoring and control
- ◆ Tool diagnostics and characterization

Space Research:

- ◆ Hazardous gas detection
- ◆ Life support and leak detection onboard Space Station
- ◆ Planetary and moon Exploration: Mars, Europa

R&D:

- ◆ Multi-sensor deployment
- ◆ Remote operation
- ◆ Particle accelerators
- ◆ Synchrotrons

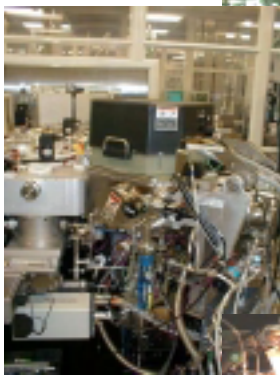
Industrial Vacuum Coating:

- ◆ Vacuum and optical coating
- ◆ Vacuum furnaces
- ◆ Glass coating
- ◆ Preventive maintenance
- ◆ Cryogenic pumps performance

Environmental Monitoring:

- ◆ Detection of volatile organic compounds (VOCs)
- ◆ Abatement monitoring
- ◆ Oceanography applications

Since it was founded in 1989, Ferran Scientific Inc. has established itself as the world leader in miniature, low cost pressure measurement and gas analysis. Using patented innovations in vacuum technology, Ferran Scientific Inc. was able to respond to pressing needs for affordable *in-situ* gas instrumentation. Upon its introduction, the Micropole mass spectrometer has enjoyed world wide acceptance and success as one of the technological breakthroughs in 30 years of quadrupole mass filter designs. The Micropole continues to be a workhorse in affordable mass spectrometry with an impressive installed base in a variety of industries. The Micropole, in its newly released **Symphony** system, is the world's smallest complete mass spectrometer system. These consumable sensor type instruments have provided lower cost of ownership in numerous applications. Using its expertise in miniaturization and mass production employing cutting edge technological tools, Ferran Scientific Inc. is able to manufacture instruments such as mass spectrometers and pressure gauges for a wide range of usage. The interchangeability of sensors and electronics adds an unprecedented plug-and-play concept for easy troubleshooting, upgrade, and replacement. The company's turnkey solutions enable the user to minimize the installation time and rapidly extract gas pressure and analysis information.



FERRAN **SCIENTIFIC**

Affordable Gas Measurement, Control and Analysis

[Http://www.ferran.com](http://www.ferran.com)
11558 Sorrento Valley rd.
San Diego, CA 92121
U.S.A

Phone: (858) 792-2332
Fax: (858) 792-0065
E-mail: sales@ferran.com

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE November 2005		3. REPORT TYPE AND DATES COVERED Final Contractor Report
4. TITLE AND SUBTITLE Identification and Quantitative Measurements of Chemical Species by Mass Spectrometry			5. FUNDING NUMBERS WBS-22-101-42-02 NAS3-01164	
6. AUTHOR(S) Mark A. Zondlo and David S. Bomse				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Southwest Sciences, Inc. 51570 Pacheco Street, Suite E-11 Santa Fe, New Mexico 87505			8. PERFORMING ORGANIZATION REPORT NUMBER E-15178	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001			10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA CR-2005-213820	
11. SUPPLEMENTARY NOTES Project Manager, Karen J. Weiland, Exploration Systems Division, NASA Glenn Research Center, organization code PTO, 216-433-3623.				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Category: 35 Available electronically at http://gltrs.grc.nasa.gov This publication is available from the NASA Center for AeroSpace Information, 301-621-0390.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) The development of a miniature gas chromatograph/mass spectrometer system for the measurement of chemical species of interest to combustion is described. The completed system is a fully-contained, automated instrument consisting of a sampling inlet, a small-scale gas chromatograph, a miniature, quadrupole mass spectrometer, vacuum pumps, and software. A pair of computer-driven valves controls the gas sampling and introduction to the chromatographic column. The column has a stainless steel exterior and a silica interior, and contains an adsorbent of divinylbenzene that is used to separate organic species. The detection system is based on a quadrupole mass spectrometer consisting of a micropole array, electrometer, and a computer interface. The vacuum system has two miniature pumps to maintain the low pressure needed for the mass spectrometer. A laptop computer uses custom software to control the entire system and collect the data. In a laboratory demonstration, the system separated calibration mixtures containing 1000 ppm of alkanes and alkenes.				
14. SUBJECT TERMS Mass spectrometers; Gas analysis; Gas chromatography; Quadrupoles			15. NUMBER OF PAGES 85	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	

